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NEW BRUNSWICK OIL SHALE/COAL ASH CHARACTERIZATION AND POTENTIAL USAGE

by

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1.0 ABSTRACT

Bulk samples of baghouse (BH) ash and fluidized bed heat exchanger (HE) ash were obtained from the circulating fluidized bed combustor (CFBC) of the New Brunswick Electric Power Commission's Chatham 22 MW demonstration plant. The ash is the product of a co-combustion trial carried out on New Brunswick Albert Formation oil shale (2 parts) and New Brunswick Minto coal (1 part) to test the effectiveness of carbonate-bearing oil shale as a sorbent for sulphur gas emissions from the high-sulphur coal.

The two ash samples, plus two mixtures of those ashes (i.e. Mix 1 comprising 1 part HE to 2 parts BH ash and Mix 2 comprising 2 parts HE to 1 part BH ash) were subjected to size analysis, mineralogical determinations, major and minor element chemical analyses, neutral and acidic leaching, moisture/density (Proctor) tests, compacted-mold hydraulic conductivity tests, freeze/thaw tests, and unconfined uniaxial compressive strength tests.

Almost 99% of the HE ash particles are greater than 75μm (200 mesh); only 11.5% of the BH ash grains are larger than 75μm. The major minerals in the ashes are quartz, plagioclase, and orthoclase feldspar, anhydrite, and magnetite. Leaching tests show that heavy metals such as As, and Pb and to a lesser degree Hg are easily leached from the HE and BH ashes under acidic conditions and from the mixed ashes under neutral conditions. Consequently, the potential for groundwater contamination must be considered in any future ash storage, industrial or construction application.

All four ash types are capable of neutralizing acid. The HE ash is the best in this regard, and has about 23% of the neutralization capacity of an equivalent amount of pure limestone ($CaCO_3$). The ash could be utilized for a low permeability cap over acid-generating mine waste such as that at Heath Steele Mines in northeastern New Brunswick.

The geotechnical tests show that: (1), all four ash types have relatively low dry unit weights that are 80% to 100% of that of loose uniform sand; (2), the compacted BH , Mix 1, and Mix 2 ashes have permeabilities similar to fine silty sand; (3), the HE ash is 60 to 90 times more permeable than the other ash types; (4), freeze/thaw cycles over a 37 day period had very little effect on compressive strength of molds of all ash types, and (5), all the ashes have pozzolanic properties. The latter characteristic would suggest that the ashes may be suitable as admixtures to low strength Portland cement concretes.

Mix 2 ash has the highest unconfined strength of the four ash types and may have an application when mixed with conventional aggregates in Portland cements or bituminous cements as a stabilizing base for highway surfaces.

1.0 RÉSUMÉ

Les échantillons en vrac de cendres de chambre des filtres (CF) et des échangeurs de chaleur à lit fluidisé (EC) ont été obtenus du combusteur à lit fluidisé (CLF) de l'usine de démonstration de 22 Mw de la Commission d'énergie électrique de Chatham. Les cendres sont le produit d'un essai de cocombustion effectué pour le schiste bitumineux du comté d'Albert du Nouveau-Brunswick (2 parties) et le charbon de Minto, Nouveau-Brunswick (1 partie) afin de vérifier l'efficacité du schiste bitumineux carbonaté à titre de sorbant pour les émissions de gaz sulfureux du charbon à haute teneur en soufre.

Les deux échantillons de cendre, ainsi que deux mélanges de ces cendres (par exemple, mélange 1 composé d'une partie de EC et 2 parties de cendre CF et mélange 2 composé de 2 parties de cendre EC et d'une partie de cendre CF) ont fait l'objet d'une analyse granulométrique, de déterminations minéralogiques, d'analyses chimiques d'éléments mineurs et majeurs, de lessivages acides et neutres, d'essais Proctor de l'humidité et de la densité, d'essais du coefficient de conductivité hydrologique, d'essais de congélation-décongélation, et d'épreuves de résistance à la compression uniaxiale non confinée.

Près de 99 % des particules de cendre de EC sont supérieures à 75 μm (200 mailles); 11,5 % seulement des grains des cendres de CF sont supérieures à 75 μm. Les principaux minéraux dans les cendres sont le quartz, le plagioclase, le feldspar orthoclase, l'anhydrite et le magnétite. Les tests de lessivage démontrent que les métaux lourds tels que le As et le Pb, et à un degré moindre, le Hg sont facilement lessivés et débarassés des cendres EC et CF dans des conditions acides et des cendres mélangées dans des conditions neutres. Il faut tenir compte de tout risque de contamination de l'eau souterraine pour tout entreposage des cendres, application industrielle ou de construction à l'avenir.

Les quatre types de cendres peuvent neutraliser l'acide. Les cendres EC sont les meilleures à cet égard, et ont environ 23 % de la capacité de neutralisation d'une quantité équivalente de pierre à chaux pure ($CaCO_3$). Les cendres pourraient être utilisées pour un revêtement de faible perméabilité des déchets de mines produisant de l'acide telles que les mines de la Heath Steele dans le nord-est du Nouveau-Brunswick.

Les essais géotechniques démontrent que: 1) les quatre types de cendre ont un poids unitaire sec relativement faible qui sont de 80 % à 100 % de celui du sable uniforme en vrac; 2) le CF compacté, les cendres du mélange 1 et du mélange 2 ont des perméabilités semblables au sable argileux fin; 3) les cendres EC sont de 60 à 90 fois plus perméables que les autres types de cendre; 4) les cycles congélation-décongélation sur une période de 37 jours ont au peu d'effet sur la résistance à la compression des moissures de tous les types de cendres, et 5) toutes les cendres ont des propriétés pouzzolaniques. Les dernières caractéristiques laisseraient supposer que les cendres peuvent être des adjuvants convenables pour les bétons de Portland de faible résistance.

Les cendres du mélange 2 ont la force non confinée la plus élevée des quatre types de cendres et peuvent avoir une application lorsqu'on les mélange avec des agrégats conventionnels dans les ciments Portland ou des ciments bitumineux comme base stabilisante pour les surfaces routières.

2.0 INTRODUCTION

On April 18, 1989 the Research and Productivity Council submitted a proposal to the New Brunswick Department of Natural Resources and Energy entitled "Oil Shale/Coal Ash Characterization and Potential Utilization". As a result, a contract was awarded to RPC on July 25, 1989 to investigate the physical, chemical and geotechnical characteristics of the combustion wastes from the New Brunswick Electric Power Commission's demonstration-scale circulating fluidized bed combustor located at Chatham N.B. The wastes for this study were a mixture of ash from the combustion of New Brunswick coal and oil shale.

The goal of the program was to characterize the CFBC wastes in order to ascertain whether the ashes have any industrial application or if saleable by-products could be derived from them. A successful application or use for the ash would reduce or eliminate disposal/storage costs.

In the event that it is not possible to determine potential uses of the ashes, or if the potential uses are not of sufficient scale to eliminate the disposal problem, consideration may need to be given to the long-term storage of the wastes. Although not part of the current work, the geotechnical data generated by the test program conducted and reported herein, will be an important data base in planning any such future storage operation.

The project consisted of three major phases:

1. Compilation of background information on the operation of the Chatham circulating fluidized bed combustion system, the generation of wastes and the associated methods of disposal. Relevant literature was examined in order to compile a summary of the characteristics of the coal and oil shale used in the process.*

2. The physical, chemical and geotechnical test program, and

3. Utilization appraisal through a literature review in order to find viable uses for the wastes.

Details of the project requirements may be found in the Terms of Reference (Appendix A, page 68).

* Contrary to the proposal assumption, no limestone was used in the test burns studied.

3.0 BACKGROUND INFORMATION

3.1 Operation of Chatham Circulating Fluidized Bed Combustion Unit [1*]

Circulating fluidized bed combustion (CFBC) systems incorporate a design which allows pulverized fuels to be burned in a suspended state brought about by an upwardly directed stream of air. This process produces a high slip velocity between the fluidized solids and combustion gases, leading to good mass and heat transfer rates and lower operating temperatures. Efficiency is consequently improved, thus permitting the use of lower ranked fuels than possible with conventional combustion units. CFBC technology also permits the easy introduction of sorbent materials, used to capture the sulphur from high sulphur coals and thereby reduce the emission of SO_2 to the atmosphere. Proper staging of primary and secondary combustion air, coupled with relatively low combustion temperatures, results in significantly lower NO_x levels as well.

NB Power's Chatham CFBC plant has a rated steam output of 95,500 kg/hr at 6,200 kPa (gauge) and 480°C with a boiler feedwater temperature of 180°C. The unit operates at a combustion temperature of 850°C.

Figure 3-1 (page 6) shows schematically the operation of the Chatham CFBC unit.

Individual weigh feeders are used to supply oil shale and coal, each crushed to a grain size <6 mm, to the combustor via elevating belt conveyor, feed bin, chain conveyor and rotary valve. When limestone is used as a sulphur sorbent, it is delivered pneumatically from a storage hopper to the combustor.

Air used for both fluidization and combustion is introduced to the combustor bottom through the nozzle grate as primary air and below the water walls as secondary air. It is this staged combustion which allows for a relatively low combustion temperature and at the same time reduces the thermal formation of NO_x.

The circulating fluidized bed combustion system is characterized by a high internal gas velocity which produces a mixture of solids and gases, with the concentration of the gases decreasing continuously from the bottom to the top of the unit. Because solids tend to leave the combustor with the flue gases in considerable amounts, a cyclone is used to separate the majority of the solids and return them to the combustor. The finer fly ash continues as part of the flue gas stream, travelling eventually through the convective pass (backpass) and baghouse, where it is then captured. Due to the high recirculation rate in the system (combustor, cyclone, fluid bed heat exchanger and combustor) the heat and mass transfer characteristics are excellent.

* Superscript numbers indicate the numbered references in the Bibliography, page 64.

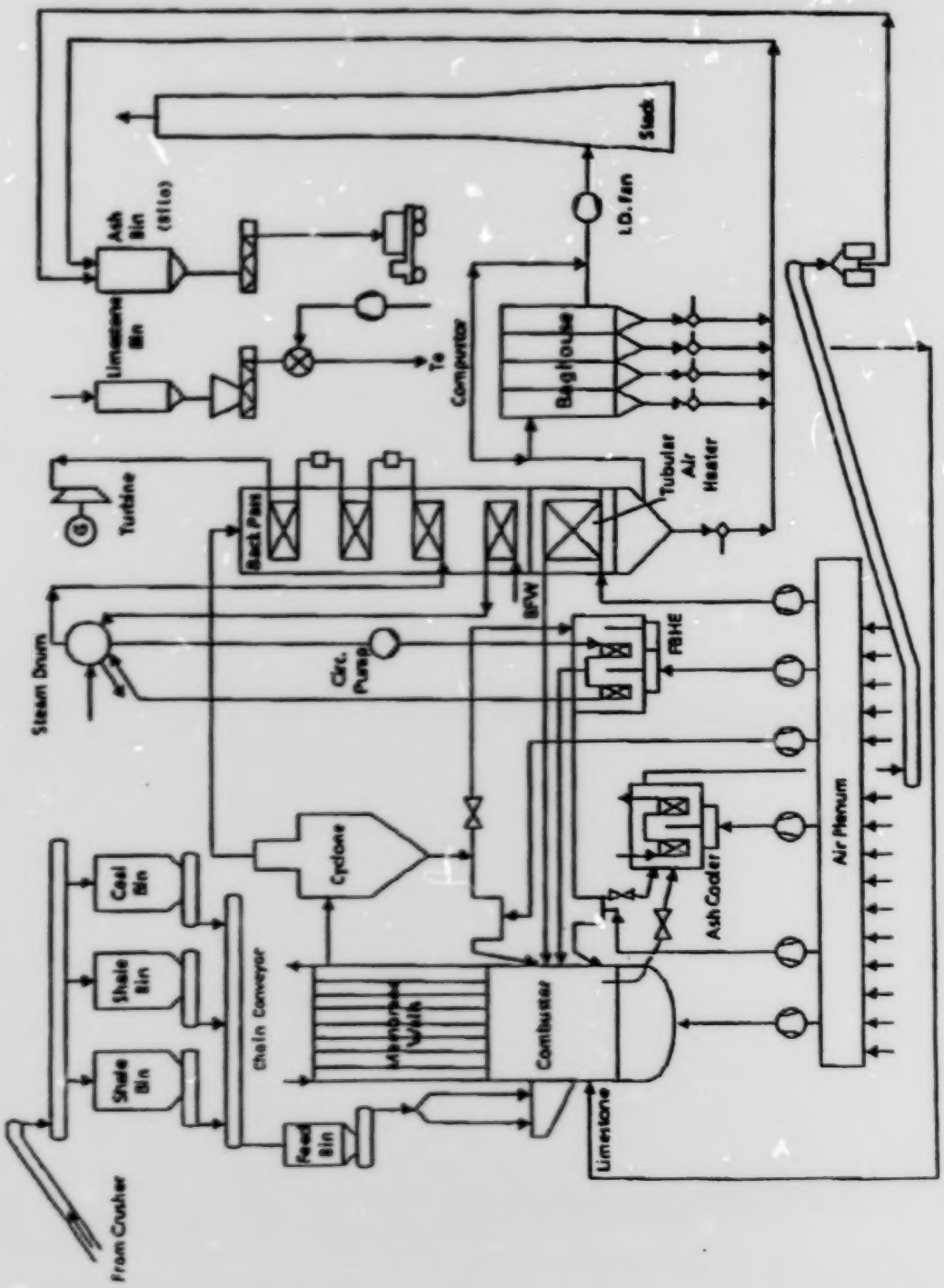

Figure 3-1: Schematic of the NBEPC Chatham CFBC Unit

The bulk of the ash discharged from the system is fly ash from the convective pass and the baghouse, which is pneumatically transported to a storage silo. It is sometimes necessary to discharge bottom ash from the system in order to maintain a desired differential pressure. This ash, which may be released downstream of the fluid bed heat exchanger (FBHE) or directly from the combustor, is conducted to a final ash cooler and then to the same silo as the fly ash.

For the combustion trials which generated the ash for this study, a 2:1 ratio of oil shale to coal was used. This figure corresponds to an actual rate of consumption of 9,090 kg/hr oil shale and 4,550 kg/hr coal. When the ash samples were collected, the boiler was operating at 50% load or 47,700 kg/hr.

3.2 Properties of Minto Coal

The coal from the Minto area of New Brunswick is sub-bituminous in rank and, as with most Eastern Canadian coals, possesses relatively high levels of both ash and sulphur. It is purchased exclusively by the New Brunswick Electric Power Commission and burned without beneficiation for the thermal generation of electricity. It is this situation which represents a potential environmental threat due to the release of SO_2 upon combustion.

Approximately 0.45 million tonnes of Minto coal is mined per year in strip operations in the Minto-Chipman area of New Brunswick. The coal occurs in a seam 20 to 40 cm thick and lies below overburden that can be as much as 30 metres thick. The coal used in the generation of the ashes studied during this project came from two areas; Coal Creek (30%) and Dragline 8200* (70%). On average, Minto coal gives an ultimate analysis as shown in Table 3-1. It is a high volatile coal with a heating value of 23-28 MJ/kg (10,000 to 12,000 Btu/lb), a sulphur content of approximately 8% and an ash content of approximately 20%.

The principal inorganic constituents of Minto coal are, as indicated in Table 3-2, iron sulphides and quartz. The former is largely pyrite and it occurs concentrated in lenses and disseminated individual grains and crystals. The quartz occurs as small grains throughout the coal. Other rock forming minerals that occur are: chlorite, illite, feldspar, gypsum, calcite, dolomite, apatite and detrital minerals such as garnet and zircon.

Semi-quantitative ICP multi-element analysis of several Minto coal samples has yielded the information in Table 3-3. Note that analyses of Dragline 8200 coal from two sources have been presented for comparison.

* Dragline 8200 is located 0.7 km off the Post Road, approximately 8 km from the Town of Minto. It is accessed by entering the Midland Strip Mine Development Area, which is operated by N.B. Coal Ltd.

Table 3-1: Ultimate Analysis of Minto Coal[1]

Constituent	Weight %
Carbon	59.9
Hydrogen	4.3
Sulphur	8.5
Oxygen	1.3
Nitrogen	0.5
Ash	20.9
Moisture	5.0

Table 3-2: XRD Mineralogy of Minto Coal[2]
(Figures shown refer to peak intensity)

Sample	Quartz	Pyrite	Chlorite	Illite	Others
Coal Creek	25	28	3	2	--
Drag 8200 *	40	35	6	1	3 (calcite)

The high ash and sulphur contents of Minto coal suggest that the potential to upgrade it by the application of suitable beneficiation techniques exists.

* Average of 3 samples; Bottom, Middle and Top of seam (30 cm thick)

Table 3-3: Semi-quantitative ICP Multi-Element Analysis of Minto Coal

Sample	Coal Creek (Wt.%)[2]	Dragline 8200 * (Wt.%)[2]	Dragline 8200 (Wt.%)[3]
SiO_2	I.S.**	I.S.**	5.18
Al_2O_3	1.81	2.28	1.60
Fe_2O_3	11.26	7.95	5.56
Mn_3O_4	--	--	--
MgO	0.08	0.26	--
CaO	0.08	0.11	0.80
K_2O	1.50	1.11	0.12
Na_2O	0.29	0.47	0.03
P_2O_5	0.03	0.13	0.32
TiO_2	0.26	0.18	0.09
SO_3	--	--	0.58
SrO	--	--	--
BaO	--	--	0.01
Fusion Loss	--	--	0.13

Sample	Coal Creek (ppm)[2]	Dragline 8200 * (ppm)[2]	
As	145	79	--
Mn	646	428	--
Zn	253	53	--
Pb	116	9	--
Cd	0	0	--
Ni	18	22	--
Ba	9	66	--
Co	72	54	--
Cu	0	1	--
Li	22	27	--
Cr	0	7	--

* Average of 3 samples; Bottom, Middle and Top of seam (30 cm thick)
** Insufficient sample

Stirling 1985 (reference 2) concluded that "The pyrites of the Minto coalfields were predominantly euhedral to subeuhedral, although framboids and finely disseminate pyrite did occur." Grain size analysis (as an area in square microns) yielded the following:

Table 3-4: Pyrite Grain Size Analysis of Minto Coal (Area in μm^2)

Coal	Mean	Median	Mode
Coal Creek	30	29	39
Dragline 8200*	26	23	15

CRL (Combustion Research Laboratory) CANMET conducted a series of pilot-scale beneficiation and combustion tests at their Devon, Alberta facility, the results of which were summarized in a 1987 report by the SNC Group [4]. They concluded the following:

> "The pilot plant tests have confirmed that Minto coal is amenable to reduction of ash content using a conventional wet cleaning technique, while the sulphur removal from this coal is limited. Even at low energy recovery of about 80%, the sulphur removal was only 41.85% which corresponded to sulphur content in clean coal as high as 4.93 wt%.
>
> Scanning electron microscope investigation has shown that Minto coal requires grinding to approximately 10 μm or less to achieve effective liberation of fine pyrite particles, which is difficult to justify as an economically viable option.
>
> Coal crushing from 100 mm to 10 mm resulted in a significant liberation of ash and sulphur from the plus 0.6 mm fraction. However, the fine fraction (0.6 x 0.15 mm) had an increased sulphur content after crushing."

3.3 Properties of Minto Coal Ash

In conventional utility boilers designed for coal burning, ash with different characteristics is trapped and collected at a number of locations within the plant. A 1986 CANMET study[5] compared the combustion performance of run-of-mine Minto coal and three washed Minto

* Average of 3 samples; Bottom, Middle and Top of seam (30 cm thick)

coals to a commercially available thermal bituminous coal from Western Canada. As a result, the major chemical components of a number of ashes were determined and have been summarized in Table 3-5. In the table, the reference to coarse or fine indicates the manner in which the parent coal was ground in preparation for each of the combustion trials. In rough terms, coarse grinding resulted in 70.0% of the coal reduced to -200 mesh, while fine grinding yielded a -200 fraction of 89.4%.

Table 3-5: Major Element Chemistry of Minto Coal Ash (Wt. %)

Major Elemental Oxides	Fly Ash		Bottom Ash		Superheater Ash	
	Coarse	Fine	Coarse	Fine	Coarse	Fine
SiO_2	29.20	27.05	36.65	37.95	25.68	28.55
Al_2O_3	11.66	11.69	15.48	17.42	8.32	8.91
Fe_2O_3	44.99	45.62	36.98	36.17	54.48	51.66
TiO_2	0.65	0.63	0.57	0.67	0.45	0.48
P_2O_5	2.45	2.52	0.05	0.04	1.38	1.49
CaO	5.72	6.34	4.22	4.45	4.53	4.74
MgO	------	0.46	0.25	0.37	0.25	------
SO_3	2.58	2.82	0.10	0.20	1.56	1.69
Na_2O	0.31	0.26	0.55	0.67	0.26	0.17
K_2O	0.83	0.81	0.53	0.58	0.49	0.49
BaO	0.32	0.38	------	------	------	0.26
SrO	------	------	------	------	------	------

Table 3-6 provides information on the trace element analysis of a fly ash sample resulting from the combustion of Minto coal at NB Power's Grand Lake generating station. The analysis was carried out at RPC as part of an in-house project to assess the utilization of this fly ash in the production of concrete. As well, the corresponding major element analysis has also been presented in order that a comparison may be made with the figures given in Table 3-5, columns 2 and 3. Note the significant difference in the reported CaO values; 5.72%/6.34% (Fly ash, Table 3-5) and 0.32% (Table 3-6).

The unusually high iron content in Minto coal ash presents a number of unique utilization problems and potentials. The average Fe_2O_3 figure from Table 3-5 is 45% while that in most other coal fly ashes is more typically in the 10%-15% range. Work has been done to concentrate this dense fraction from the ash for use as a substitute for conventional magnetite used as a dense media in coal beneficiation plants. Trials at RPC in 1986[7]

Table 3-6: Major and Trace Element Analysis of Minto Coal Ash[6]

Constituent	Wt. %
SiO_2	29.85
Al_2O_3	10.50
Fe_2O_3	49.30
CaO	0.32
MgO	0.06
Na_2O	1.32
K_2O	1.44
Ignition Loss	4.23

Constituent	Wt.*
Li	8
Be	<1
Na	3.550 %
Mg	1.240 %
Al	6.900 %
K	0.100 %
Ca	1.020 %
Mn	0.040 %
Fe	3.890 %
Rb	<8
Sr	60
Nb	9
Ba	<1
Ta	<8
V	128
Cr	5

Constituent	Wt.*
Co	18
Ni	12
Cu	3
Zn	16
As	18
Se	<5
Mo	<1
Ag	<0.5
Cd	<1.0
Sn	<10
Sb	<5
Te	<10
W	<10
Pb	18
Bi	<2
U	<10

* ppm unless otherwise indicated

showed that it was possible to produce a magnetic fraction from Minto fly ash which had a specific gravity of 3.9 and heavy media properties similar to natural magnetite. In fact, the fly ash concentrate (FAC) was less abrasive than magnetite, stayed in suspension longer, and possessed the same pH in slurry.

This work also suggests the possibility of using the fly ash, now devoid of most of its iron, as a pozzolan for concrete admixtures. Such a scenario, although not investigated, could represent a high-volume usage which is currently not possible due to the high iron content of the ash.

Although iron is a problem in many concrete applications, it has been demonstrated that it is possible to substitute up to 30% Minto fly ash for cement and still attain strengths which are 60% of those achieved by standard concrete mixes[8]. At the 20% level of substitution, the pozzolanic activity index (PAI) was above 75% which renders the concrete acceptable for certain high strength applications. At greater levels of substitution, and

therefore lower strength levels, there could be a market for this concrete in back-fill applications, particularly in the mining industry[9].

3.4 Properties of Albert Oil Shale

Oil shale occurs in Albert, Westmorland and to a lesser extent in Kings County in southeastern New Brunswick. The most organic-rich and thickest beds which are exposed at the surface occur at Albert Mines and Rosevale in Albert County and in the Dover and Saint-Joseph areas of Westmorland County.

The oil shale beds range up to 20 metres or more in thickness and give yields of 75 to 125 litres of oil per tonne of rock[10]. Oil shale beds at all surface exposures and in drill hole intersections are seen to be interbedded with less organic-rich shale beds and barren shales, siltstones and minor sandstones, limestones, dolostones and conglomerates. The highest-grade (most organic-rich) shales comprise alternating microlaminations of kerogen - the organic complex - carbonate minerals and silicates. The microlaminated character of the shale gives the rock one of two habits. First, in those beds where the laminations are not crenulated or folded, the shale weathers and splits into paper thin sheets. Second, where the shale is flow folded and fresh it tends to be tough and blocky. The less organic-rich beds have fewer kerogen laminae and the carbonate and silicate laminae are correspondingly more abundant and thicker.

The relatively high content of carbonate minerals in the oil shales (particularly calcite and dolomite) has led some writers to refer to the shales as marlstones or dolomitic marlstones[11a,11b]. Siderite is a minor carbonate phase. In addition to carbonates, these shales contain detrital quartz, feldspar, clay minerals and minor pyroxene (Table 3-7). Analcite is a common accessory and is believed to have formed from the alteration of clay minerals by alkali-rich waters such as reported by Berry and Mason[12] for the Green River oil shales.

The Albert formation is divided into a number of members, based on composition. The lower unit is predominantly sandstone, siltstone, shale and conglomerate and is known as the Dawson Settlement Member. A medial unit, the Frederick Brook Member, is comprised of oil shale, limestone, calcareous shale and siltstone, while the upper, or Hiram Brook Member is siltstone, shale, calcareous sandstone and minor oil shale.

The Frederick Brook member is in turn divided into four lithographic units, as described by Macauley and Ball[11b]. The three lower units, in ascending order, the dolomite marlstone zone, the clay marlstone zone, and the Albert Mines zone, consist primarily of dolomitic marlstone, clay marlstone and laminated marlstone, respectively. In the upper unit, or Upper zone, shale, dolostone and dolomite marlstone predominate.

Wright[11c] carried out semi-quantitative XRD mineralogical determinations on 145 samples of Albert Formation rocks (Table 3-7). The samples Wright analyzed are from drill core, and represent three holes drilled by Petro-Canada in Westmorland County in 1982. Most, but not all of the samples are from oil shale beds. The automated XRD method employed characterized the shape and relative peak-area intensity of the diagnostic peak of each mineral and compared that peak to quartz which was assigned an intensity factor 1.00. In Table 3-7 the character of the diagnostic peak, the mean value, and the standard deviation for the major identified mineral phases are given. The shales clearly contain abundant clay (illite and kaolinite), carbonate minerals (calcite, dolomite, and siderite), and quartz and feldspar.

Table 3-7: Mineralogical Summary and Correlation of Albert Oil Shale

	Shale Type		
	Dover 1 (42 samples)	Dover 2 (86 samples)	Boudreau 1 (17 samples)
Illite	moderate 17.8±8.0	moderate 19±8.3	high 28±9.6
Kaolinite	high 7±3.5	variable 2.2±2.7	low 1.9±0.9
Chlorite	high 1.8±1.1	erratic,low 0.41±0.48	low, erratic 0.27±0.23
Analcite	erratic, high 5±3.0	variable 2.4±2.2	low, erratic 0.72±0.72
Pyroxene	erratic 1.4±0.6	consistent 1.2±0.6	low 0.89±0.58
Quartz	16±4.7	16±3.2	16±1.9
Feldspar	high 30±9.8	high 26±6.2	low, variable 18±7.0
Calcite	consistent 6±4.3	erratic 6.6±5.9	erratic, high 9.4±7.7
Dolomite	erratic,low 13±10.4	consistent, high 25.6±8.7	high 26.4±13.4
Siderite	erratic 0.54±0.32	high, consistent 1.65±1.2	erratic, low 0.73±1.0
Pyrite	erratic 3.4±2.4	consistent, high 3.4±1.2	low, 1.7±0.62
Ill/Kao	very low 1.2±1.2	high 11.7±13.5	moderate 7.3±4.9
Qtz/Fsp	consistent 1.6±0.7	erratic 1.8±0.6	erratic, high 2.9±1.2
Cal/Dol	moderate, variable 4.0±7.2	erratic, very low 0.7±0.9	high, variable 20.5±58

Table 3-8 gives the average major[11d] and minor[11e] element chemistry of the oil shale. The major constituents are based on the average of three samples containing 25 Imperial gallons of shale oil per ton. These oil shale samples were provided to the Research and Productivity Council by the New Brunswick Department of Natural Resources as part of a project to evaluate the design concepts and economics for coal/oil shale combustion retort systems. The minor elements are averages of 14 samples obtained from the Frederick Brook Section of the Albert Formation.

Because these figures are derived by different analytical techniques on different samples from different areas, they cannot be reconciled in all cases. For example, CaO is listed at 10.75% and calcium as 14.29% (142,857 ppm).

Finally, ultimate analyses of Albert oil shale are shown as Table 3-9[11f]. The Upper Shale Zone, Albert Mines Zone, Clay Marlstone Zone and the Dolomite Marlstone Zone in this table refer to the four lithotypes of the Frederick Brook Member of the Albert Formation, described at the beginning of this section.

Table 3-8: Major and Minor Element Chemistry of Albert Oil Shale

Element	Wt.%	Element	ppm
SiO_2	40.78	lithium	67
Al_2O_3	12.87	vanadium	25
Fe_2O_3	5.20	rubidium	41
CaO	10.75	magnesium	33,700
K_2O	1.61	aluminum	37,929
Na_2O	1.28	calcium	142,857
P_2O_5	<0.01	beryllium	5
TiO_2	0.37	copper	35
MnO	0.12	lead	54
MgO	3.58	zinc	84
Loss on Ignition	21.62	silver	3.35
		nickel	32
Element	ppm	molybdenum	24
		uranium	7.7
strontium	943	gold	0.13
fluoride	644	% phosphate	0.16
boron	0.16	gallium	N.D.
chromium	40	barium	102

Table 3-9: Ultimate Analysis of Albert Oil Shale (Wt.%)

	Upper Shale Zone 1	Albert Mines Zone 2	Clay Marlstone Zone 3	Dolomite Marlstone Zone 4
Organic Carbon	2.75	7.40	6.18	7.25
Inorganic Carbon	3.07	3.45	2.97	1.70
Hydrogen	0.83	1.26	1.07	1.05
Nitrogen	0.21	0.15	0.07	0.24
Sulphur	0.77	0.82	0.73	0.67
Oxygen	10.67	10.48	9.98	9.25
Ash	81.70	76.44	79.00	79.84

3.5 Properties of Albert Oil Shale Ash

Major element chemistry for Albert oil shale ash is presented as Table 3-10[11f], carbon analyses are shown in Table 3-11[11f] and minor element chemistry in Table 3-12[11g]. Once again, the four zones indicated in Table 3-10 refer to the zones of the Frederick Brook Member of the Albert Formation described previously. Analysis consisted of one sample per zone.

Test samples 1, 2, 3, and 4 (Table 3-11) are samples of retorted oil shale (not combusted oil shale) taken under four different sets of oil shale retort operating parameters[11f]. Consequently, the analyses are not directly comparable with the CFBC ashes being assessed in this study. Again, one sample of fresh shale blend and one sample from each test were analyzed.

Table 3-10: Major Element Chemistry of Albert Oil Shale Ash (Wt. %)

	Upper Shale Zone 1	Albert Mines Zone 2	Clay Marlstone Zone 3	Dolomite Marlstone Zone 4
SiO_2	44.86	50.86	51.02	51.34
Al_2O_3	24.48	19.64	20.39	21.58
Fe_2O_3	7.79	7.91	8.01	8.08
CaO	10.22	11.84	9.26	8.10
K_2O	3.43	2.26	2.32	2.43
Na_2O	3.60	1.72	2.40	2.65
MgO	5.62	5.77	6.60	5.82

Table 3-11: Carbon Analysis of Fresh Albert Oil Shale and Oil Shale Ash*

Carbon Wt%	Fresh Shale Blend	Spent Shale From Tests 1	2	3	4
Total carbon	9.34	2.20	4.06	3.42	2.41
Organic carbon	6.72	0.91	1.91	1.71	0.93
Inorganic carbon	2.62	1.29	2.15	1.71	1.48

* The fresh shale blend referred to in Table 3-11 consisted of a mixture of the following lithotypes: the Albert Mines Zone, the Clay Marlstone Zone and the Dolomite Marlstone Zone. Due to the insufficient quantities available, Upper Shale was not included in the blend.

Table 3-12: Minor Element Chemistry of Albert Oil Shale Ash (Average of 14 Samples - Wt. %)

	Vanadium	Nickel	Molybdenum	Copper
Shale Ash	0.026	0.004	0.006	0.008

4.0 TEST PROGRAM AND RESULTS

The test program was undertaken via sample preparation, ash size analysis, ash mineralogy, chemical analyses, leaching tests, hydraulic conductivity tests, freeze/thaw tests, and compressive strength tests, each of which is described in the following sections.

4.1 Sample Preparation

Two, 45-gallon drums of combustor ash were received from the Chatham circulating fluidized bed combustion plant. One contained 137 kg of FBHE (fluidized bed heat exchanger) ash, while the other contained 102 kg of BH (baghouse) ash.

Each drum was mechanically rolled for four hours to ensure thorough mixing of the ash. The contents were then emptied into two separate stock piles from which a 70 kg sample of FBHE ash and a 34.5 kg sample of BH ash were taken. Following this, two mixed samples were prepared. Mix 1 was formed from 1 part FBHE ash to 2 parts BH ash, by weight, to give a sample of 67.5 kg. Mix 2 was prepared from 2 parts FBHE ash to 1 part BH ash with an aggregate weight of 67.0 kg. Mix 1 and Mix 2 were reloaded into drums and mechanically rolled for four hours to effect homogenization.

Five kilogram samples of each of the four ashes were then taken for chemical analysis and leach testing. The remainder was returned to four individual drums for use during the rest of the test program.

4.2 Size Analysis

Each sample was run through a series of nine Tyler screens (meshes 6, 10, 20, 60, 100, 200, 270, 325, 400) using a Tyler Ro-Tap sieve shaker. The amount of material retained on each screen was determined and the weight percentages calculated. The amount of ash passing the 200 mesh screen in each case was also determined. When this figure equalled or exceeded 50% of the total weight of the sample, the minus 200 fraction was subjected to further classification using a Warman cyclosizer.

The Tyler screen size analyses, as well as the -200 mesh cyclosizer analyses are presented in Table 4-1 and graphically in Figure 4-1.

More than 50 percent by weight of the BH and Mix 1 ash is in size fractions less than 200 mesh. Consequently, both of these ashes were further classified using the Warman cyclosizer. The cyclosizer figures given are percentages of the -200 mesh fraction obtained from the screen analysis, not percentages of the entire ash sample.

Table 4-1: Size Analysis by Tyler Screen and Warman Cyclosizer

Screen Size Mesh	μm	HE Ash (%)	BH Ash (%)	Mix 1 (%)	Mix 2 (%)
+6	+3360	6.83	0.00	1.22	11.94
+10	+2000	9.11	0.00	1.49	2.44
+20	+840	18.30	0.00	1.92	5.80
+60	+250	37.68	0.19	18.63	33.19
+100	+150	15.36	0.49	1.98	4.10
+200	+75	11.51	10.98	12.34	14.36
+270	+53	0.41	21.58	14.16	11.19
+325	+45	0.12	14.57	15.55	10.03
+400	+37	0.26	13.44	10.76	5.03
-400	-37	0.42	38.75	21.95	1.92
TOTAL		100.00	100.00	100.00	100.00
-200 mesh		1.21	88.34	62.41	28.17
Cyclone 1	+32	-	45.91	53.39	-
Cyclone 2	+29	-	14.63	13.47	-
Cyclone 3	+23	-	16.71	15.07	-
Cyclone 4	+16	-	11.88	10.35	-
Cyclone 5	+09	-	6.22	4.67	-
	-09	-	4.65	3.05	-
TOTAL			100.00	100.00	

The HE ash is the coarser of the two parent ashes with approximately 99% falling in the +200 mesh range. The BH ash on the other hand, is finer with only 12% greater than 200 mesh. More than 87% of the HE ash is larger than 100 mesh; only 0.68% of the BH ash is larger than 100 mesh.

Not surprisingly, the Mix 1 ash, which was made up of two parts BH ash and one part HE ash, exhibited size distribution characteristics much like the BH ash. Cyclosizer analysis of the BH and Mix 1 ashes reveal virtually identical fraction weights (Table 4-1), although the BH ash does contain a somewhat greater proportion of finer material.

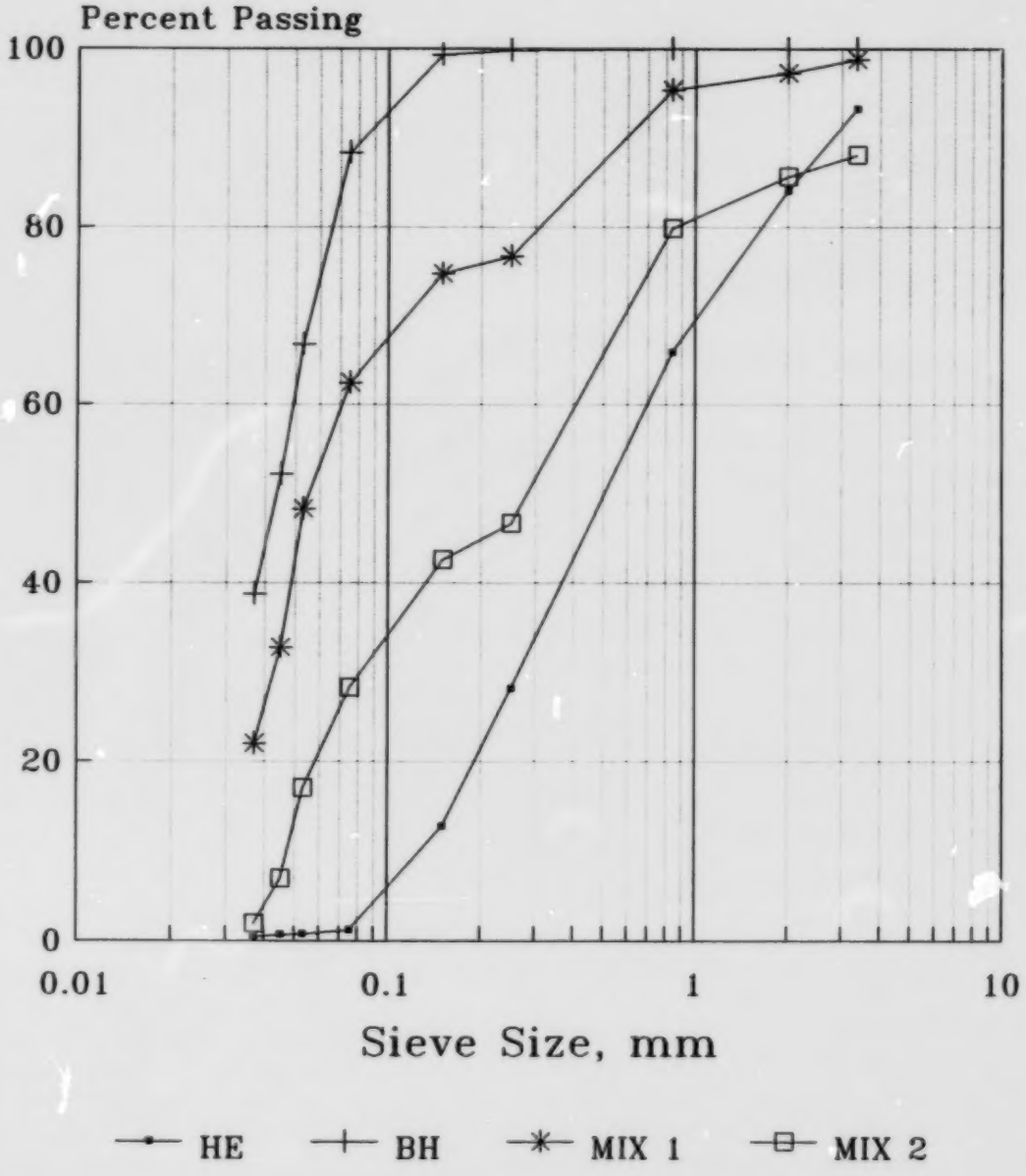

Figure 4-1: Size Analysis of Ashes

4.3 Mineralogy

The four ash samples were examined by stereo microscope and by electron microprobe analyzer (EPMA) in order to identify the major mineral phases present. This information was complemented by submitting pulverized samples of the ashes to x-ray diffraction (XRD) techniques. From this it was possible to assign semi-quantitative percentage values to the mineral constituents. In addition, observations on ash morphology were recorded where appropriate.

The major minerals of each ash (those which were identified by both EPMA and XRD) were:

1. quartz
2. sodium-aluminum silicate (albite)
3. sodium-calcium-aluminum silicate (plagioclase)
4. potassium-aluminum silicate (possibly orthoclase)
5. anhydrite
6. magnetite

In addition to these minerals, there were also some accessory phases which were noted using the EPMA but not detected by x-ray diffraction. They were:

7. calcium compound (probably oxide)
8. magnesium-iron silicate
9. iron-titanium oxide
10. calcium-sulphur-iron compound

In order to calculate the relative volume percentages of the major mineral phases it was first necessary to assume that they accounted for the entire volume of each sample. This was not unreasonable, as the percentages were determined by examining the intensities of the XRD peaks (see Appendix B). The secondary constituents were not detected by XRD at all, indicating that their contribution to the whole was minimal. Table 4-2 summarizes the mineralogical data.

Particle shapes in the four ashes varied from irregular to sub-rounded. Spherical forms, such as cenospheres, were very rare.

Very little amorphous material was detected in any of the ash samples. As well, there was minimal evidence of high temperature products, ie melted and recrystallized quartz.

All of the ash samples showed evidence of some anhydrous phases, specifically anhydrite.

Table 4-2: Volume Percentages of the Major Mineral Phases

Component	Sample			
	HE	BH	Mix 1	Mix 2
Quartz	48	51	49	43
Na-Al silicate	8	8	11	9
Na-Ca-Al silicate	10	5	5	8
K-Al silicate	12	9	6	12
$CaSO_4$	12	16	18	16
Fe_3O_4	10	11	11	12
Total Sample	100	100	100	100

The clay minerals, such as illite, kaolinite and chlorite (Table 3-7), were no doubt modified by the high temperatures encountered during the combustion process; the primary change being the release of water from their structures. The temperatures were presumably high enough to destroy the clay mineral structure but not high enough to form glass. The other components of the clays (SiO_2, Al_2O_3 and K_2O) are still evident however. Table 4-3 presents a chemical analysis of illite and kaolinite.

Table 4-3: Chemical Analyses of Illite and Kaolinite[13]

Component	Illite (Wt.%)	Kaolinite (Wt.%)
SiO_2	46.81	45.80
Al_2O_3	36.09	39.55
Fe_2O_3	---	0.57
CaO	0.29	0.41
K_2O	10.24	0.03
Na_2O	0.68	---
P_2O_5	---	---
TiO_2	0.01	---
MnO	---	---
MgO	0.62	0.14
H_20^+	5.00	13.92
H_20^-	0.42	0.17

A comparison of the information contained in Tables 4-2 and 4-3 indicates that the major constituents of illite (SiO_2, Al_2O_3 and K_2O) and the major constituents of kaolinite (SiO_2, Al_2O_3) have been retained in the K-Al Silicate/Na-Al silicate complexes identified by XRD examination.

4.4 Chemical Analyses

The objective of the chemical analyses was to determine the weight percentages of various oxides (SiO_2, Al_2O_3, $FeO*Fe_2O_3$, CaO, MgO, MnO, Na_2O, K_2O, P_2O_5 and TiO_2), total organic carbon (TOC) and a number of metals and trace elements (Cu, Pb, Zn, As, Ag, Li, Ba, V, Cr, Cd, Ni, Mn, Sr, Rb, Hg and U). The primary method of analysis was ICAP (inductively coupled argon plasma), although Ti, Li, Ba, V, Sr and Rb were done by AA (atomic absorption), Hg by flameless AA, As by the Gutzeit method, TOC by Leco furnace, and P colorimetrically.

It was suspected that the ashes might contain free lime and/or calcium sulphide, two components which could have significant bearing on storage or utilization practices. Free lime was therefore determined by sucrose/water dissolution followed by titration against oxalic acid with a phenolphthalein indicator. Calcium sulphide content was obtained by converting to the sulphate for measurement, and then subtracting from total sulphur. At the same time, the neutralization capacities of the ashes were determined, in order to assess the potential of the ash in neutralizing acid mine drainage. Neutralizing capacity is calculated by first converting sulphur in the sample to equivalent H_2SO_4 (sulphuric acid), which yields the theoretical acid production. The capability of the ash to consume acid is then determined by titrating it with 1.0N sulphuric acid until a pH of 3.5 is reached. The volume of acid used is recorded. Typically, this procedure can take several days to several weeks as the sample continues to generate a base. Finally, the loss on ignition was determined for each of the ashes in order to ascertain if any unburnt carbon was present.

The results of the chemical analyses of the four ashes are given in Table 4-4. It will be noted that the sums of the constituents do not equal 100% in any of the four samples analyzed. Under oxidizing conditions of combustion, a number of refractory oxides can form which are not easily leached. Consequently, the usual digest method, which employs a mixture of bromic, hydrochloric and nitric acids to release silica, did not affect this insoluble fraction of the ashes. The samples were therefore analyzed again with HF in the digest and a total insolubles fraction of 8% - 10% was determined in each case. The insolubles are likely to be mixtures of Al_2O_3, $FeO*Fe_2O_3$, CaO and MgO, with the first two constituents accounting for the bulk of the material.

SiO_2 is the most abundant constituent of each ash, averaging 51%. CaO is the next most prevalent oxide in the HE ash, comprising 12% by weight, while $FeO*Fe_2O_3$ (at 9.73%) is the second most common in the BH ash. Al_2O_3 and MgO, both in the range of 5%-8.5%, account for most of the remaining oxides. Apart from the 1.202% K_2O found in the HE ash, all other constituents are present in amounts which are less than 1%.

The neutralization capacities of the four ashes are given in Table 4-5.

Table 4-4: Chemical Analyses of Ashes

	HE Ash %	BH Ash %	Mix 1 %	Mix 2 %
SiO_2	51.09	50.71	50.74	51.67
Al_2O_3	8.45	6.01	6.54	6.80
$FeO*Fe_2O_3$	6.01	9.73	8.09	6.85
CaO	12.02	8.75	10.07	10.88
MgO	6.56	4.62	5.30	5.84
MnO	0.12	0.11	0.12	0.12
Na_2O	0.53	0.40	0.43	0.44
K_2O	1.202	0.801	0.755	0.712
P_2O_5	0.26	0.52	0.26	0.52
TiO_2	0.17	0.12	0.13	0.12
Cu	0.005	0.008	0.01	0.005
Pb	0.015	0.016	0.016	0.013
Zn	0.01	0.01	0.01	0.01
As	0.10	0.10	0.08	0.09
Ag (ppm)	<0.10	<0.10	<0.10	<0.10
Li	<0.05	<0.05	<0.05	<0.05
Ba (ppm)	<0.001	<0.001	<0.001	<0.001
V	0.014	0.014	0.019	0.021
Cr	0.009	0.009	0.009	0.009
Cd	<0.001	<0.001	<0.001	<0.001
Ni	0.0045	0.0066	0.0059	0.0050
Mn	0.091	0.087	<0.092	0.090
Sr	<0.05	<0.05	<0.05	<0.05
Rb	<0.005	<0.005	<0.005	<0.005
Hg	0.010	0.009	0.009	0.009
U (as UF) (ppm)	3.5	3.1	3.6	3.4
S	1.50	2.30	2.08	1.63
SO_4	1.49	2.06	2.07	1.16
PO_4^{-3} (ppm)	1.1	<0.5	1.3	2.8
NO_3^{-1} (ppm)	14	24	20	18
Free Lime (as CaO)	0.7	0.6	0.6	0.8
TOC	0.15	1.46	0.98	0.52
Loss on Ignition	5.63	1.43	2.43	3.77
TOTALS	96.25	89.99	90.95	92.19

Table 4-5: Neutralization Capacities

Ash	% S	Theoretical Acid Production kg/tonne (A)	Acid Consumed kg/tonne (B)	A - B	Acid Producer
HE	1.50	45.89	219.67	-173.78	No
BH	2.30	70.36	111.38	-41.02	No
Mix 1	2.08	63.63	150.73	-87.10	No
Mix 2	1.63	49.86	169.00	-119.14	No

Based upon the presence of sulphur in the ashes, the theoretical acid production was calculated*. The figures in Table 4-5 are an indication of the potential for acid production by these ashes if, for example, they were to be landfilled and thereby exposed to water. Titration with 1.0N sulphuric acid was then undertaken to determine the ability of the ashes to consume acid as a consequence of their alkali content. The A-B column indicates that the ashes all have the ability to neutralize any acid that they themselves might generate, as well as additional acid that might be present in the environment. The HE ash has the highest neutralization capacity at 219.67 kg/tonne while the BH ash is the poorest at 111.38 kg/tonne, presumably because of its lower CaO content.

4.5 Leaching Tests

After examination of a number of leaching scenarios and discussion with the Environmental Protection Service (EPS) of Environment Canada it was decided, as indicated in the Terms of Reference for this project (Appendix A), to make use of the equilibrium leach test (ELT) protocols currently being evaluated by Environment Canada. This procedure was developed by the Wastewater Technology Centre, Canada Centre for Inland Waters, in Burlington Ontario. A copy of the methodology is included in Appendix C.

The method specifies that the test material be minus 100 mesh and so it was necessary to grind all except the Baghouse (BH) ash. Samples of 160 g of each ground ash were leached in solutions of (a) deionized water, and (b) acidic water with a pH of 4.5 prepared from hydrochloric acid. The leaching was carried out for a period of 7 days, under fixed agitation at room temperature. Upon completion of leaching, the slurry was filtered and the resultant

* Theoretical Acid Production (lb/ton) = % S/100 * 3.059 * 2000 where 3.059 is the molecular weight of H_2SO_4 divided by the molecular weight of S (98.081/32.064). Multiply by 0.5 to get kg/tonne (kg/1000 kg).

cake thoroughly washed. To ascertain if a weight loss had taken place, the wash water and leachate were combined for analysis and the total dissolved solids (TDS) determined. In addition to the TDS, the following elemental and ionic concentrations were determined: Na, K, Ca, Pb, Hg, As, SO_4^{-2}, PO_4^{-3}, NO_3^{-1}, and H^+ (ie pH).

The results of the Equilibrium Leach Tests (ELT) carried out on the four ashes are summarized in Table 4-6.

Table 4-6: Results of Chemical Analysis of Leachates From Equilibrium Leach Tests

Test Description	As (mg/L)	Ca (mg/L)	Hg (mg/L)	K (mg/L)	Na (mg/L)	Pb (mg/L)	TDS (mg/L)	pH	SO_4^{-2} (mg/L)	PO_4^{-3} (µg/L)	NO_3^{-1} (mg/L)
(1) HE (pH 7.0 Leach)	1.01	1192	<0.001	104.0	51.55	1.16	3544	9.53	1750	<5	3.4
(2) HE (pH 4.5 Leach)	2.84	788.1	<0.001	73.25	29.58	3.73	3561	9.57	2080	<5	N.D.*
(3) BH (pH 7.0 Leach)	2.35	601.0	<0.001	113.4	35.99	3.05	2829	10.10	1600	<5	2.8
(4) BH (pH 4.5 Leach)	3.03	473.1	<0.001	81.34	24.01	4.10	2935	10.17	1580	<5	N.D.
(5) M1 (pH 7.0 Leach)	3.60	6.44	(0.001	29.51	9.70	4.93	2982	9.51	1600	<5	2.6
(6) M1 (pH 4.5 Leach)	2.93	1.75	<0.001	23.52	8.15	4.11	2919	9.55	1800	<5	N.D.
(7) M2 (pH 7.0 Leach)	2.87	1.80	<0.001	22.81	7.91	4.02	3078	9.61	1560	<5	2.4
(8) M2 (pH 4.5 Leach)	2.10	1.30	<0.001	19.12	6.20	3.03	3069	9.66	1620	65	N.D.

* N.D. = Not determined

Ca, K, and Na are more soluble in the neutral leach than in the acidic (pH 4.5) leach for all ash types. Arsenic and lead values are higher for the HE and BH ashes than for the mixes under acidic leaching. This trend is reversed for the mixtures, where the neutral leach liberates more As and Pb. The amount of mercury obtained from each sample is consistently <0.001 mg/L regardless of the leaching pH employed.

The TDS (total dissolved solids) data reveal a situation similar to that found for arsenic and lead. Levels are very slightly higher for acidic leaching of HE and BH ashes, but are lower for the two mixes. All TDS values fall within the 2800-3600 mg/L range with the highest values recorded for the HE ash leach tests. The next highest levels are found for the Mix 2 ash, not surprisingly, as it comprises 2 parts HE ash to 1 part BH ash.

The leaching chemistry is controlled by such factors as common ions, electrical charge of species in solution, ionic strength of the solution, formation of complexes, and pH. Considering all these variables plus the differences in grain size and chemistry of the HE and BH ash (Table 4-4), it is not unexpected to find reversals in ionic solubility between parent and mixed ashes.

The pH of the resulting leachates are in every case slightly lower for the neutral leaches than for the pH 4.5 leaches, although for each ash, the similarity of pH is evident.

Sulphate concentrations are fairly similar within each pair of tests, with the higher value generally resulting from the pH 4.5 leach. Phosphates are all <5μg/L (except Mix 2, acid leach - 65 μg/L) regardless of the ash type or the leaching regime employed, while nitrate levels are 2.4 mg/L to 3.4 mg/L.

The fact that the sums of the ionic species do not equal the TDS values is to be expected. In order to achieve a balance, all ions present in solution would have to be analyzed. This situation is discussed further in Section 6.2.

4.6 Moisture/Density Relationship Tests

Moisture/Density Relationship tests were carried out on each of the four ash sample types, using the standard ASTM D 698-78 Method A (See Appendix D). This method is utilized for material passing the No. 4 (4.75 mm) sieve and employs a four inch (101.6 mm) diameter mold.

The specimens were prepared by placing the thoroughly mixed ash in three layers of approximately equal height and compacting each layer with 25 blows of the hammer - a 2.49 kg rammer dropped 305 mm. The ASTM procedure requires that at least four specimens of each sample at varying moisture contents be prepared. Four such specimens proved sufficient for the two "mixture" samples. Additional data points were obtained for the bag-house (BH) and heat exchanger (HE) ashes for determination of the maximum dry densities and optimum moisture contents.

The results of moisture/density determinations on compacted specimens of ash are shown graphically in Figure 4-2. Maximum dry unit weights, optimum moisture contents and solid mineral grain specific gravities are given in Table 4-7. Also shown are porosity, void ratio and degree of saturation values applicable at the maximum dry unit weights.

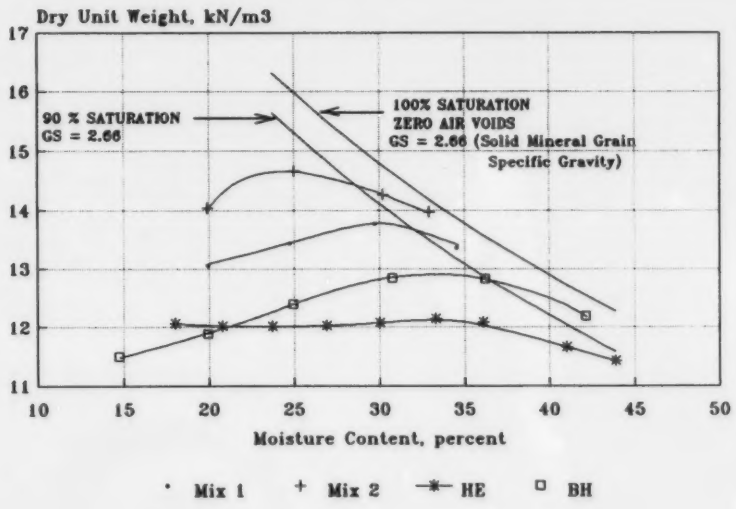

Figure 4-2: Moisture/Density Relationships, Dry Unit Weight vs Moisture Content

Mix 2 (2HE:1BH) is the most dense of the four ashes, when compacted at its optimum moisture content. It has a dry unit weight, at its maximum value, broadly comparable with that of a loose uniform sand. The lightest material, molded from the heat exchanger (HE) ash, has a unit weight of approximately 80 or 90 percent of that of a typical loose sand or 65 percent of that of a typical compacted sand and gravel.

Table 4-7: Moisture/Density Relationships

Material Type	Maximum Dry Unit Weight, kN/m^3	Optimum Moisture Content, %	Porosity at Maximum Unit Weight n	Void Ratio at Maximum Unit Weight e	Degree of Saturation at Maximum Unit Weight %	Mineral Grain Specific Gravity, Gs
BH	12.9	33	0.51	1.02	86	2.66
Mix 1 (1HE:2BH)	13.8	30	0.47	0.89	89	2.66*
Mix 2 (2HE:1BH)	14.7	25	0.44	0.78	86	2.67*
HE	12.2	34	0.53	1.15	79	2.67

* Based on the relative proportions of each parent ash.

4.7 Hydraulic Conductivity Tests

Hydraulic Conductivity tests were performed on recompacted samples of each of the four ash types. Hydraulic conductivity varies with grain size and with grain size distribution. Since air bubbles within a soil sample behave like impenetrable solid grains, the hydraulic conductivity tends to be lower in unsaturated soils than in water-saturated ones. It is therefore customary to saturate the sample either prior to, or in the course of laboratory testing.

Based on their grain size distributions, the hydraulic conductivities (K) of the baghouse ash and to a lesser degree the mixtures containing this ash, were expected to be relatively low. A back-pressure permeameter is generally utilized for ensuring saturation of very fine grained samples. The method involves application of a back-pressure to the sample which has the effect of compressing air bubbles, eventually resulting in their dissolution. The back-pressure apparatus was not utilized for the conductivity tests for three reasons. Firstly, the time required for saturation would have been prohibitive, particularly for the finer baghouse

ash, and would in any case vary from one ash to the next. Secondly, the back-pressure permeameter is of metal construction which might have interfered with the chemistry of recovered leachate, particularly for low pH permeants. Finally, and most importantly, the back-pressure arrangement is a closed system which is not conducive to recovery of leachate.

A constant-head permeameter, shown schematically in Figure 4-3, was therefore constructed to carry out the hydraulic conductivity tests. The apparatus was made of acrylic material which permitted visual observation of each sample during prolonged testing, and the recovery of leachate on a daily basis. Photographs of the apparatus are presented in Figures 4-4a and 4-4b. The testing began with each sample in an unsaturated condition, corresponding, approximately, to the optimum moisture content at which it was compacted. During the test, the moisture content rose to approach the saturated value.

The four samples were tested simultaneously, with each receiving permeant from the same reservoir (ensuring the same chemistry) kept at the same constant head. Downward flow was maintained through each sample and air bubbles were periodically released at the base when required, particularly in the first ten days or so. The hydraulic gradient through each sample was held constant by a lower constant head cell (Figure 4-3). The gradients were adjusted such that the flow rates through each sample were similar. To achieve this, the hydraulic gradient across the relatively porous heat exchanger (HE) ash was very much less than that maintained across the fine baghouse (BH) ash.

The tests began using deionized water. After approximately five days, sulphuric acid was introduced to the water so that the influent pH was reduced to approximately 3.1. To achieve this, between two and three millilitres of acid were added per 20 litres of deionized water. The target pH was 4.5 but it later became apparent that 3.1 was consistently the pH that had been achieved. Hydraulic conductivities were determined, and a 250 ml leachate sample was recovered daily.

Leachate samples were refrigerated prior to the chemical analysis of selected batches. For each ash, the first pore volume of distilled water leaching, the first pore volume of acidic leaching and the final pore volume of acidic leaching were analyzed. Additionally, in the case of the HE ash, the final pore volume of distilled water leaching (after 8 days) was analyzed as was a middle volume (after 24 days) of the acidic leach tests. It was felt that this extra information would be useful to more accurately define the leaching process.

The results of hydraulic conductivity tests carried out on each of the four ash types are summarized in Table 4-8.

The effective size, D_{10}, and the Uniformity Coefficient U (or D_{60}/D_{10}), for these ashes is shown in Table 4-8. Ten percent by weight of constituent particles are finer than the effective (D_{10}) size while sixty percent are finer than the D_{60} size. These parameters are presented because the hydraulic conductivities of uniform fine soils can generally be predicted from them. Hazen[14], for example, showed that the hydraulic conductivity of a

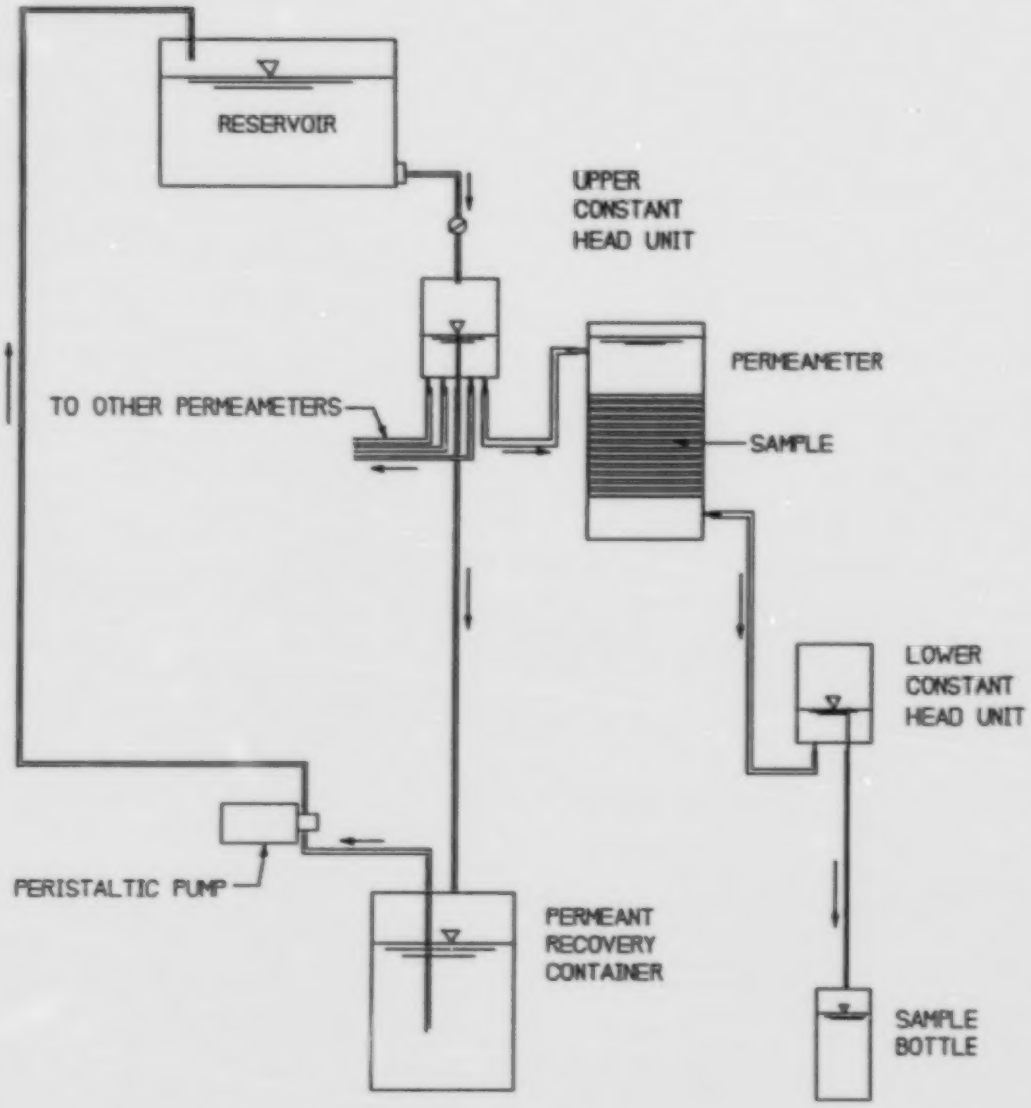

Figure 4-3: Schematic of Hydraulic Conductivity Test Apparatus

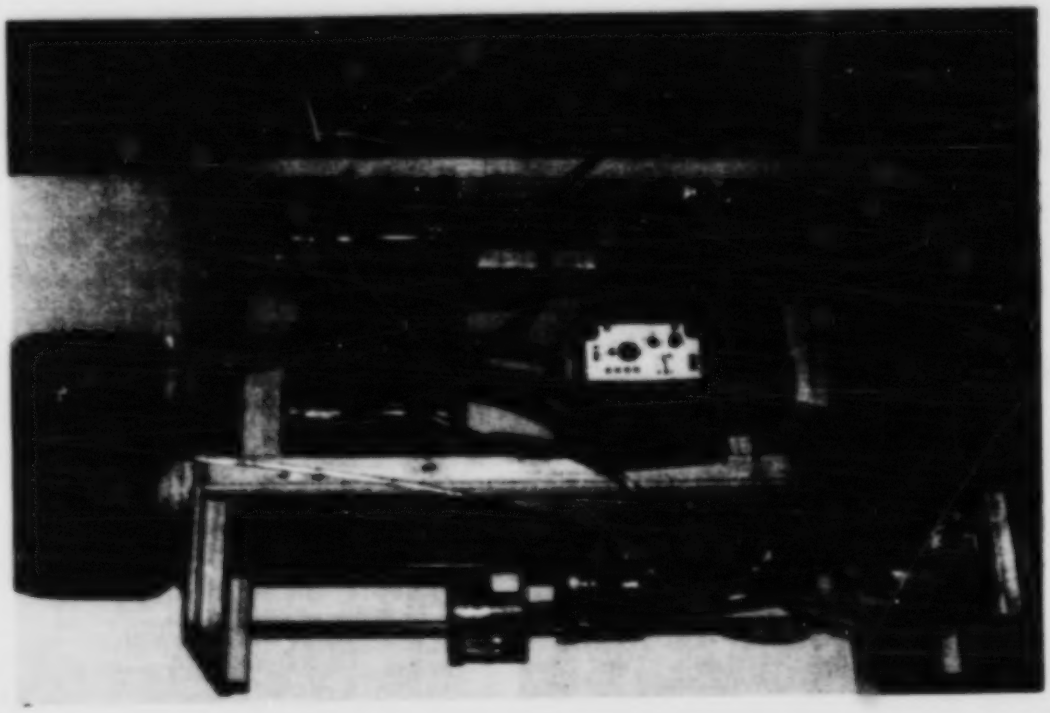

Figure 4-4a: Photograph 1 of Hydraulic Conductivity Test Apparatus

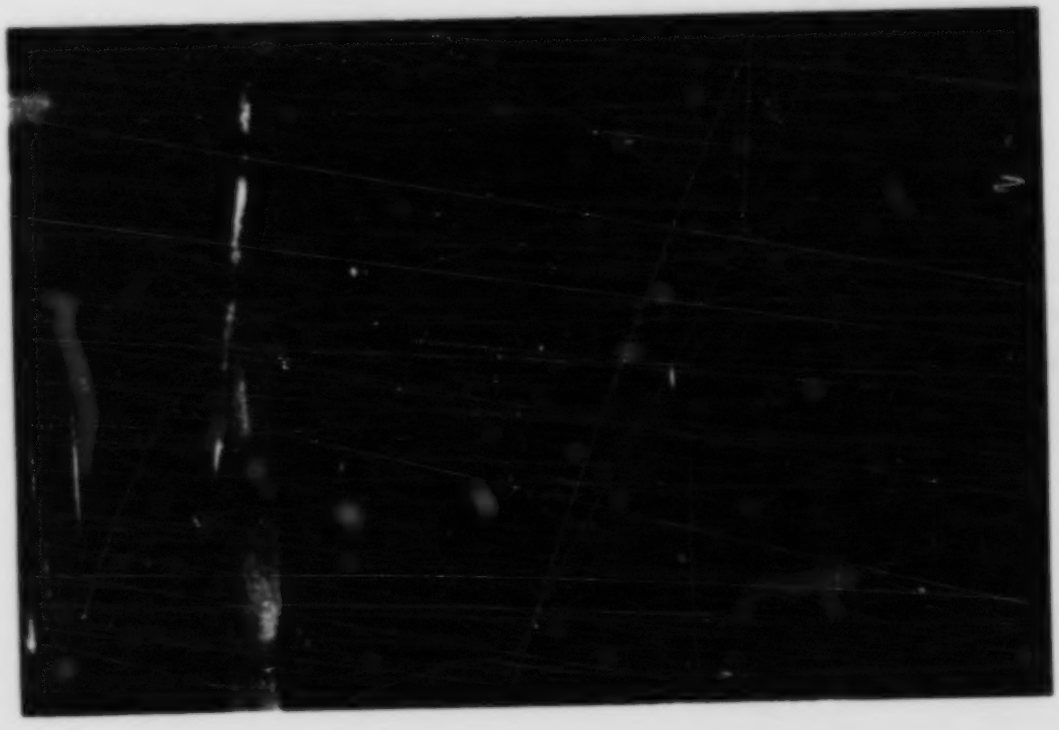

Figure 4-4b: Photograph 2 of Hydraulic Conductivity Test Apparatus

highly uniform fine sand could be estimated from the following empirical equation:

$$K\ (cm/s) = C_1 \times D_{10}^2$$

where D_{10} is the effective size in centimetres and C_1 [1/(cm x s)] is a constant varying from about 100 to 150.

From Table 4-8 it can be seen that the Hazen relationship over-estimates the hydraulic conductivity of the finer ashes, although on the basis of sample uniformity, a reasonable correlation might have been anticipated. This may be attributable to the probable difference in grain shape between the ashes and natural sands. In addition, electrochemical processes are likely to be more active for the ashes than would be the case for inert, fine sands.

For practical purposes the BH ash and both mixtures comprising BH material, have very similar hydraulic conductivities, at approximately 1×10^{-4} cm/s. The mixtures are slightly less permeable than the BH ash, presumably because their less-uniform size distribution led to reduced porosity even though they comprised coarser sizes.

The hydraulic conductivity of the HE ash is on the order of 60 to 90 times higher than that of the BH ash or of the two mixtures, and, although still lower, is more closely comparable to that anticipated for a sand of a similar gradation.

The experimental arrangement (Figures 4-3, 4-4a, 4-4b) permitted the hydraulic conductivities of each sample to be determined daily for the 50 or so day duration of the test; the progress of each sample in this regard being illustrated graphically in Figures 4-5, 4-6, 4-7 and 4-8. For non-pozzolanic materials, hydraulic conductivities normally rise as the test proceeds because the degree of saturation increases with time. Dissolution of the mineral fabric with time, and an associated increase in porosity, would presumably produce the same effect. Pozzolanic effects would be expected to produce the reverse trend, that is to say, cause a reduction in hydraulic conductivity with time, if part of the pore space is replaced with cementitious material.

For the bag house (BH) ash and for both ash mixtures (Mix 1 and Mix 2) the hydraulic conductivities rose with time to the values reported in Table 4-8. Further into the test, hydraulic conductivities gently declined with passage of additional pore volumes, or in the case of Mix 2, appeared to level off. The reduction in hydraulic conductivity, where it occurs, is probably attributable to a reduction in porosity due to pozzolanic effects. This may be the process responsible for the behaviour of the coarsest (HE) ash. This ash does not exhibit the initial rise in K normally associated with sample saturation, perhaps because saturation occurs much more rapidly.

The number of pore volumes at which the pH of the permeant was reduced to 3.1 is shown for each sample in Figures 4-5 through 4-8. Reduction of permeant pH appears to have had little effect on hydraulic conductivity with time.

Table 4-8: Summary of Hydraulic Conductivity Test Data

Sample	D_{10} mm	Uniformity Coefficient (U) (D_{60}/D_{10})	K, Hazen cm/s	K, Experimental cm/s	Porosity[2] n
BH	0.017	2.6	3.6×10^{-4}	1.0×10^{-4}	0.55
Mix 1 (1HE:2BH)	0.022	3.3	6.1×10^{-4}	6.7×10^{-5}	0.47
Mix 2 (2HE:1BH)	0.047	8.5	N/A[1]	8.6×10^{-5}	0.49
HE	0.13	5.4	N/A[1]	6.0×10^{-3}	0.58

(1) Too high a uniformity for Hazen relationships to be applicable
(2) Porosity = volume voids/total volume; n = 0.55 is equivalent to 55%

After completion of the hydraulic conductivity tests, the samples were extruded and their moisture contents determined. Each sample exhibited some degree of cementation, qualitatively described in Table 4-9 (last column). Table 4-9 also gives the wet and dry unit weights and the moisture contents and porosities of each specimen at the start and end of the testing period. All samples were essentially saturated at the end of the tests. Each specimen experienced a loss in solids weight as a result of dissolution; the losses being as much as 10 percent. The losses are reasonably consistent with those calculated from the Total Dissolved Solids (TDS) of selected leachate samples. Total Dissolved Solids were generally elevated in those leachate samples recovered immediately after the permeant had been acidified.

The reduction in dry unit weight due to mineral dissolution, and the associated increase in porosity, do not cause these samples to disintegrate with time. On the contrary, all ashes exhibited some degree of cementation after testing. It appears that new minerals formed by cementation are less soluble than the indigenous mineral grains.

The results of the chemical analysis performed on selected leachates generated during the hydraulic conductivity tests are given in Table 4-10. The first pore volumes of both the distilled water leach (day 1 or day 1+2) and the acidified water leach (day 13, 14 or 16), plus the last pore volume of the acid leach (day 53), were tested. In the case of the HE ash tests, two extra leachate samples were analyzed over that requested in the terms of reference (Appendix A). A sample of the last pore volume of the distilled water leach (day 8) and a middle volume from the acid leach (day 24), were also examined.

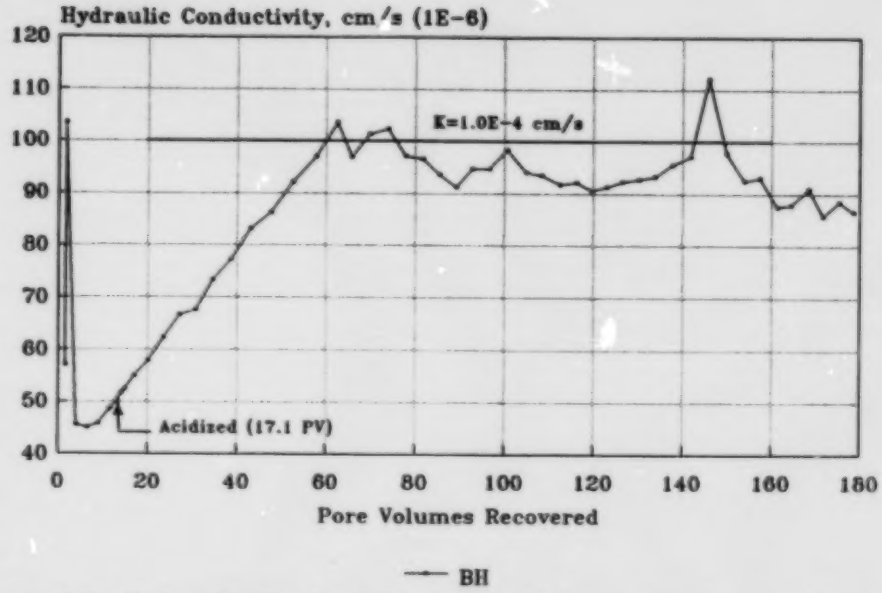

Figure 4-5: Hydraulic Conductivity vs Pore Volumes Recovered - BH Ash

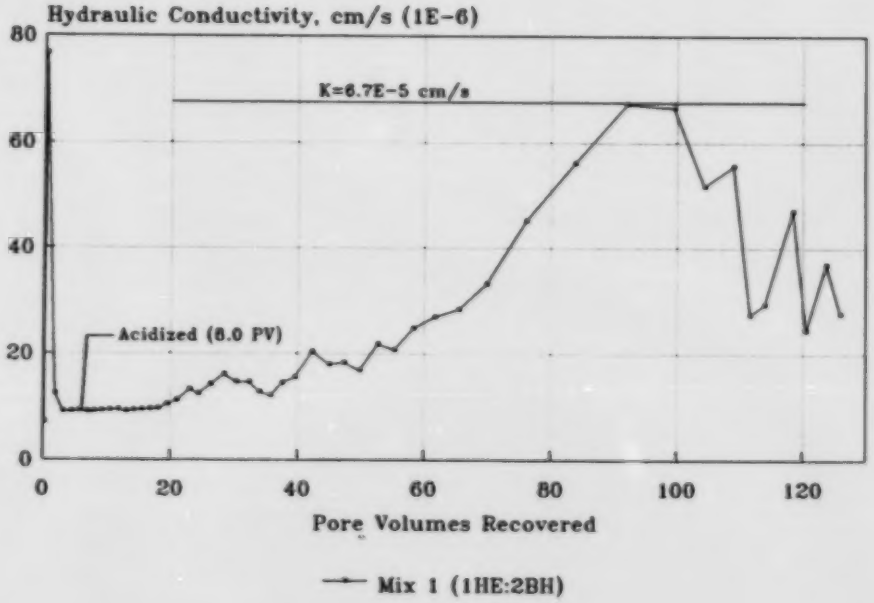

Figure 4-6: Hydraulic Conductivity vs Pore Volumes Recovered - Mix 1 (1HE:2BH)

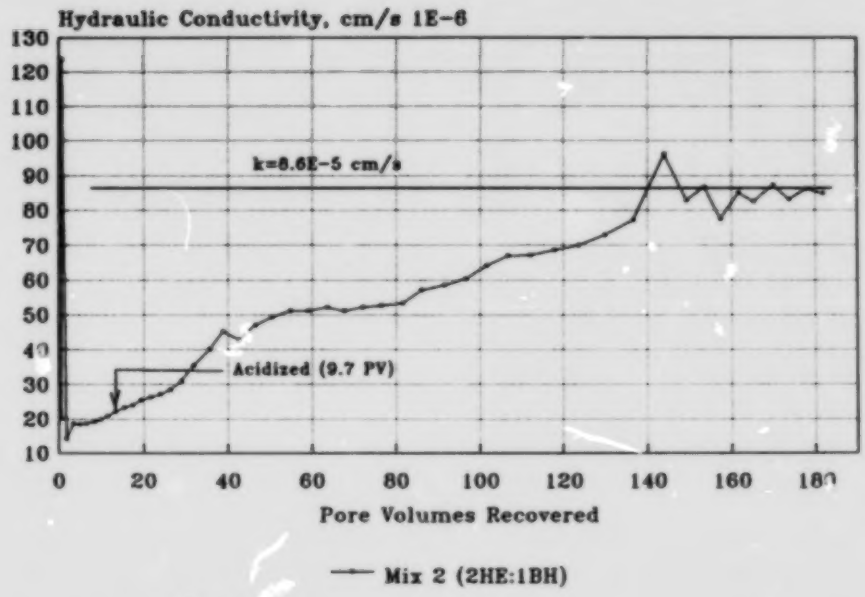

Figure 4-7: Hydraulic Conductivity vs Pore Volumes Recovered - Mix 2 (2HE:1BH)

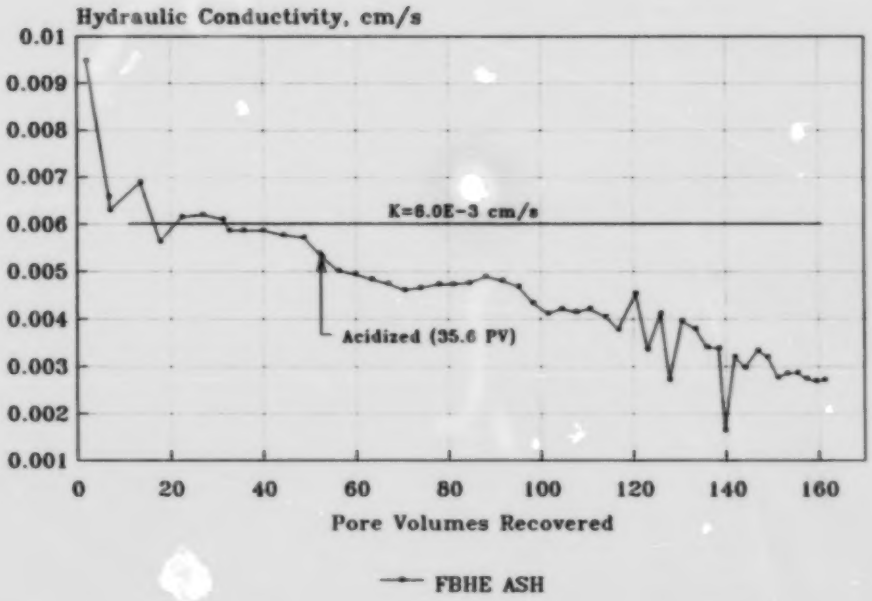

Figure 4-8: Hydraulic Conductivity vs Pore Volumes Recovered - HE Ash

Table 4-9: Characterization of Hydraulic Conductivity Specimens Before and After Passage of Permeant

	Initial Condition					Final Condition				
Material	Wet Unit Wt kN/m³	Dry Unit Wt kN/m³	Moisture Content %	P	Degree of Saturation %	Wet Unit Wt kN/m³	Dry Unit Wt kN/m³	Saturated Moisture Content %	P	D of C
BH	15.57	11.63	33.9	0.55	72.6	17.63	10.16	59.0	0.61	Moderate
Mix 1 (1HE:2BH)	17.83	13.74	29.8	0.47	88.0	19.17	12.57	40.5	0.52	Moderate-High
Mix 2 (2HE:1BH)	16.64	13.25	25.6	0.49	70.2	18.57	11.75	46.0	0.55	High
HE	14.90	11.07	34.6	0.58	67.6	16.97	10.00	60.7	0.62	Very Low

P = Porosity D of C = Degree of Cementation

The pH of the initial pore volume of the distilled water HE leachate (day 1) is 11.11, considerably more basic than that from the corresponding BH ash test. Further, the concentrations of the leached elements, with the exception of the heavy metals, are higher than those encountered with the BH ash. For example, Ca, TDS and sulphate levels are 9, 9.2 and 8.2 times higher than those from the BH ash. The heavy metals, As, Hg and Pb leached to a lesser degree; 4.9, 3.6, and 10.1 times less than those from the BH ash.

Comparison of the final pore volume (day 8) of the distilled water test, relative to that of the first pore volume (day 1), indicates that heavy metal leaching had accelerated. Arsenic concentrations increased by 2.8 times, mercury by 4.1 times and lead by 4.0 times. TDS levels held essentially constant, sulphate increased by 16% and calcium by 25%. The concentrations of both potassium and sodium decreased by approximately 4.5 times. By the start of the acidified hydraulic conductivity tests (day 13), leaching of heavy metals had again increased, as had the levels of potassium and sodium. All other ionic species, pH and TDS showed a decrease.

Near the midpoint of the HE ash tests (24 days) the levels of all leached species showed noticeable decreases, and by the time of the last volume analysis (day 53), they had dropped still further, with the exception of sodium which increased somewhat.

The results of the HE ash tests show clearly its ability to neutralize acid. For example, the pH of the solution at the end of the distilled water tests (day 8) was 10.88. The pH of the first acidified pore volume (day 13) rose to 11.00, from where it fell to 10.84 and then to 10.25 in the last volume of fluid tested. As established in Section 4.4, all of the ashes have the ability to neutralize a specific quantity of acid (Table 4-5) and it is this ability which gives rise to the basic leachates.

Table 4-10: Results of Chemical Analysis of Leachates From Hydraulic Conductivity Tests

Ash Type	Day of Sample	As (mg/L)	Ca (mg/L)	Hg (mg/L)	K (mg/L)	Na (mg/L)	Pb (mg/L)	TDS (mg/L)	pH	SO_4^{-2} (mg/L)
HE	1	0.146	669.5	0.065	89.7	35.2	0.095	2744	11.11	1850
HE	8	0.407	840	0.264	20.7	7.5	0.381	2692	10.88	2150
HE	13	0.686	556	0.349	28.8	11	0.925	1977	11.00	1300
HE	24	0.577	130.6	0.229	21	8.1	0.913	403	10.84	154
HE	53	0.29	75.69	0.02	10.89	9.32	<0.1	276	10.25	115
BH	1+2	0.714	74.8	0.237	18.0	14.5	0.964	298	8.52	226
BH	13	2.67	925.5	1.09	19.5	7.0	4.09	3089	10.09	2320
BH	53	0.07	73.26	0.01	13.98	9.62	<0.01	150	10.11	85
Mix 1, 2BH:1HE	1+2	1.18	97	0.377	30.5	24.5	1.97	412	7.71	288
Mix 1, 2BH:1HE	16	2.02	924	0.866	31.5	12.4	3.16	3030	10.25	2240
Mix 1, 2BH:1HE	53	0.07	322.8	0.02	34.76	16.31	<0.1	1235	10.21	775
Mix 2, 1BH:2HE	1+2	1.17	183.4	0.394	57.45	24.03	1.84	763	9.89	538
Mix 2, 1BH:2HE	14	1.61	920.5	0.764	33.55	33.6	2.31	3031	10.70	2460
Mix 2, 1BH:2HE	53	0.04	195.7	0.02	17.66	11.06	<0.1	666	10.24	450

Analyses of the BH ash leachates were performed on the first pore volume collected from the distilled water leaching (days 1+2), the first pore volume from the acidified leaching (day 13), and finally from the last pore volume from acidified leaching (day 53). In these samples, leaching was more vigorous during the first day of acidic leaching than it was during the first day of distilled water leaching. Most ionic concentrations increased substantially, with the exception of potassium which only changed from 18.0 mg/L to 19.5 mg/L and sodium which actually decreased from 14.5 mg/L to 7.0 mg/L. By the time of the last acidic sample, the leaching process had diminished to a level less than that encountered during the distilled water tests. The first sample (days 1+2) had a pH of 8.52, the second (day 13) 10.09 and the last one (day 53), 10.11.

The Mix 1 ash (2BH:1HE) treated with distilled water (days 1+2) shows species concentrations somewhat higher than their BH test counterparts due to the influence of one part HE ash in the mixture. A lower initial pH of 7.71 characterized this sample.

As previously, the switch to acid leaching for Mix 1 resulted in increased concentrations of leachable elements in the first pore volume (day 16), with the only exception being sodium which decreased from 24.5 mg/L to 12.4 mg/L. Total dissolved solids and sulphate levels increased in a manner similar to that encountered with the BH ash; they rose by 7.4 and 7.8 times respectively. At the same time, the solution became more basic and the pH increased to 10.25.

By the time the final volume of Mix 1 leachate was collected after 53 days, the leaching process had subsided considerably. The pH remained stable at 10.21, calcium, TDS and sulphate concentrations dropped by a factor of three, and heavy metal leaching declined 30 to 40 times. Potassium and sodium levels showed little change.

Note that in both the BH and Mix 1 tests the initial pH was somewhat lower than in the other two ashes. Examination of Table 4-5 shows that the BH ash has the lowest acid neutralization potential of the four samples. This fact is reflected in the low pH for day 1+2 leachate from the BH and Mix 1 ashes.

Analyses of the first pore volume of distilled water for Mix 2 (days 1+2) were much like those encountered with the HE ash. This was not unexpected as Mix 2 contains two parts of HE ash to one part BH ash. The BH ash component did inhibit the leaching activity as evidenced by the generally decreased levels of leached species. TDS and sulphate concentrations were more like those of the BH ash, and the pH level was between that found in the BH and HE leachates.

Commencement of acid leaching (day 14) produced the expected increase in leaching activity. TDS and sulphate levels rose from 763 mg/L and 538 mg/L to 3031 mg/L and 2460 mg/L respectively and pH increased to 10.70. The concentration of heavy metals in solution increased by up to 90%, calcium levels increased five times, potassium levels declined by a factor of two and sodium levels rose marginally.

By day 53, the leaching activity had deceased noticeably, with every parameter showing a decline. pH dropped to 10.24 and heavy metal concentrations fell by 23 to 40 times. TDS and sulphate concentrations declined to a level less than that of the first distilled water pore volume.

4.8 Freeze/Thaw Tests

Freeze/thaw tests were carried out in accordance with ASTM D 560-82 (see Appendix E). Specimens were prepared utilizing a specially manufactured four inch (101.6 mm) diameter split mold. Ash placement and compaction within the mold were as described in ASTM D 698-78 Method A (See Section 4.6 Moisture/Density Relationship Tests and Appendix D). The split mold permitted sample extrusion with minimal disturbance.

For soil-cement mixtures, for which the ASTM D560-82 method was developed, two specimens are prepared for each material to be evaluated. One specimen, termed the soil-cement loss specimen, is brushed between freezing cycles. However, given the loosely consolidated nature of the ash samples immediately after compaction, it was decided to omit the brushing stage. Two sets, comprising a total of twenty-four freeze/thaw specimens, were prepared. One set of twelve specimens was permitted to soak-up additional moisture prior to the first freezing cycle as per ASTM D 560-82, while a second set of twelve was frozen immediately after extrusion from the compaction molds. Each set comprised six specimens of Mix 1 and six specimens of Mix 2. The freeze/thaw test results are summarized in Table 4-11. Associated compressive strength data are presented in Section 4.9.

Most specimens lost weight as a result of the freeze/thaw activity; this being attributed to a loss of solids by attrition and a loss of moisture, presumably in the freezing cycle. Figures 4-9 through 4-12 show the progress of each test. The weight loss over the 15 cycle test period was typically 2 to 3 percent for the moist cured samples and 2 to 6 percent for the non-moist-cured ones. Mix 1 may be slightly more susceptible to weight loss than Mix 2, but this was not positively established.

Table 4-11: Freeze Thaw Test Data - Weight Changes With Freezing Cycles

Specimen No.	Sample Type	Type	Age, Days	No. Cycles	Weight Loss at end of test, %
28	Mix 1, 1HE:2BH		17	5	0.65
29	Mix 1, 1HE:2BH		17	5	-1.03
30	Mix 1, 1HE:2BH		17	5	0.95
31	Mix 1, 1HE:2BH		37	15	3.02
32	Mix 1, 1HE:2BH		37	15	0.57
33	Mix 1, 1HE:2BH	Moist Cured 7 Days	37	15	2.17
34	Mix 2, 2HE:1BH		17	5	1.45
35	Mix 2, 2HE:1BH		17	5	0.05
36	Mix 2, 2HE:1BH		17	5	0.85
37	Mix 2, 2HE:1BH		37	15	1.45
38	Mix 2, 2HE:1BH		37	15	-0.35
39	Mix 2, 2HE:1BH		37	15	2.07
40	Mix 1, 1HE:2BH		10	5	2.35
41	Mix 1, 1HE:2BH		10	5	1.90
42	Mix 1, 1HE:2BH		10	5	1.80
43	Mix 1, 1HE:2BH		30	15	5.20
44	Mix 1, 1HE:2BH		30	15	4.61
45	Mix 1, 1HE:2BH	No Moist Curing	30	15	5.57
46	Mix 2, 2HE:1BH		10	5	3.09
47	Mix 2, 2HE:1BH		10	5	-0.14
48	Mix 2, 2HE:1BH		10	5	2.97
49	Mix 2, 2HE:1BH		30	15	1.96
50	Mix 2, 2HE:1BH		30	15	-1.53
51	Mix 2, 2HE:1BH		30	15	2.62

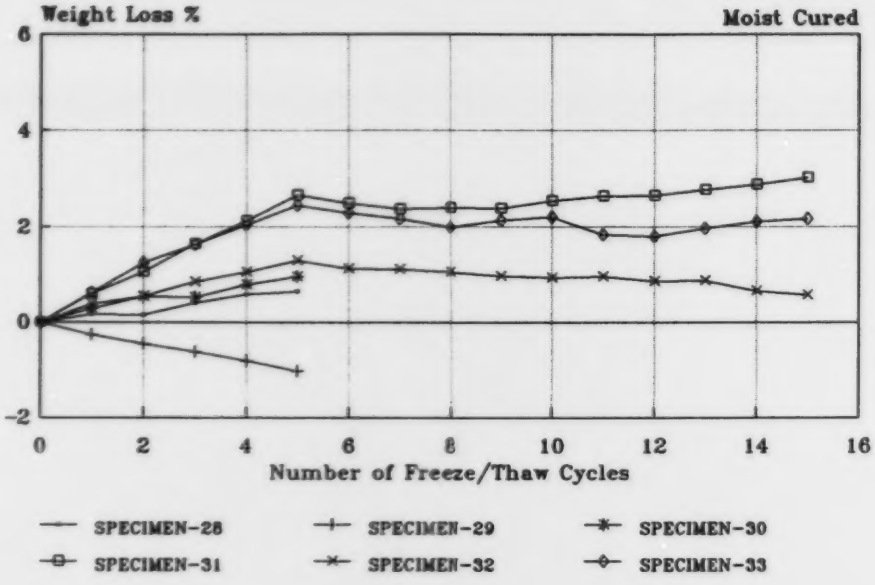

Figure 4-9: Weight Change vs No. of Freeze/Thaw Cycles - Mix 1 - Moist Cured

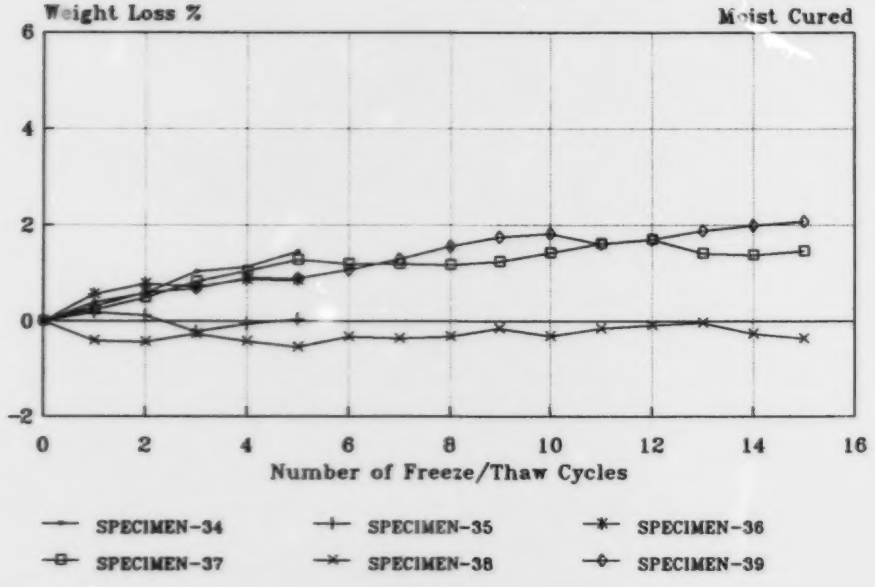

Figure 4-10: Weight Change vs No. of Freeze/Thaw Cycles - Mix 2 - Moist Cured

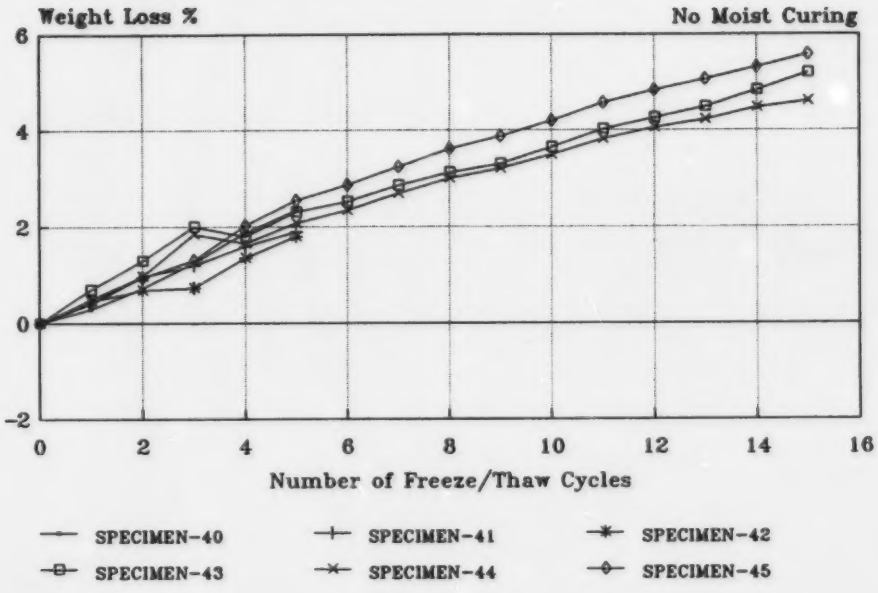

Figure 4-11: Weight Change vs No. of Freeze/Thaw Cycles - Mix 1 - No Moist Curing

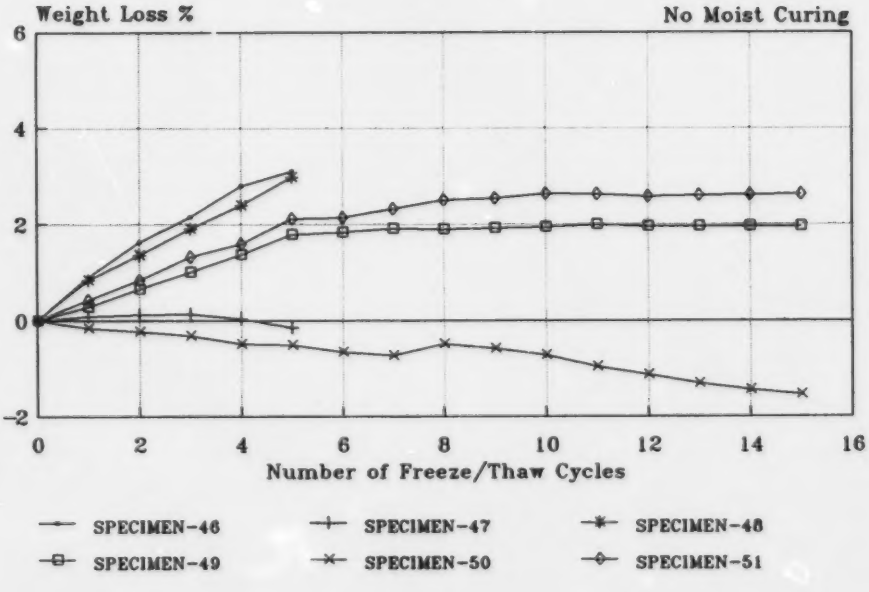

Figure 4-12: Weight Change vs No. of Freeze/Thaw Cycles - Mix 2 - No Moist Curing

From a quantitative viewpoint, the weight loss data are difficult to interpret, and may indeed be very difficult to reproduce. Subtle changes in the design of refrigerated samples boxes or the configuration of samples within them, for example, may affect the results. Note that the Mix 1 - Moist-Cured samples (Figure 4-9) ceased to lose weight significantly at 5 cycles when the shorter term specimens were discarded and the number of molds per box therefore halved. Qualitatively, however, the freeze/thaw tests indicate that these ashes, when properly compacted, maintain their integrity when subjected to freeze/thaw cycling. Field behaviour in a freeze/thaw environment would depend on access to water and on the rate of temperature change.

4.9 Compressive Strength Tests

The unconfined compressive strength of mold specimens was determined with a Tritest 50 loading frame, an appropriate proving ring for measurement of the applied load, and suitable dial gauges for the measurement of deformation. The tests were carried out in accordance with the standard ASTM D 1633-84 procedure - "Compressive Strength of Molded Soil Cement Cylinders" (see Appendix F). The loading rate adopted for the tests was 1 mm per minute, corresponding to a specimen strain rate of approximately 1 percent per minute.

Compressive strengths were determined by uniaxially loading to failure a total of 73 specimen molds. The strengths of each of the four ash types were evaluated. In the case of Mixed ashes 1 and 2, the variations in strength with time and with freeze/thaw cycling were also determined.

Table 4-12 shows compressive strength data for each of the four sample types, at a variety of initial moisture contents and therefore dry unit weights. These specimen molds were those produced for the moisture/density relationship tests (Section 4.6). Figure 4-13 is a reproduction of the moisture density relationship graph presented in Figure 4-2 but includes, for each data point, the unconfined compressive strength, in kPa.

The heat exchanger (HE) ash specimens proved to have insufficient apparent cohesion to be extruded for uniaxial testing. For these specimens, compressive strengths were evaluated by means of a penetrometer.

Figure 4-14 illustrates that for each material type, the unconfined compressive strength was at its maximum at a moisture content significantly below optimum; a normal phenomenon for conventional mineral soils[15].

Table 4-13 shows the results of supplementary compressive strength tests carried out on each of the four moist cured ash materials at various ages. These data are plotted graphically in Figure 4-15.

Immediately after mold formation, the compacted BH ash mixtures had very low compressive strengths in the 100 to 200 kPa range. At this very early stage the HE ash strength was too low for uniaxial testing. After seven day moist curing, the strengths had

risen to 50 kPa for the HE material and 300 to 700 kPa for the others. After 20 or so days, Mixes 1 and 2 had similar compressive strengths; these being about 25 percent lower than the 900 kPa of the BH ash. With further time, strengths continued to rise but Mix 2 gained strength at an increasing rate. Two months after mold preparation the compressive strength of Mixes 1, and 2 had reached 1700 and 3100 kPa respectively while the BH ash strength was intermediate between these two values. Although like the others, the HE specimens gained strength with time, the 540 kPa value achieved after two months had been surpassed by the other ashes at ages of between 1 and 2 weeks. For comparison, conventional soil-cement mixtures have unconfined compressive strengths of 2 to 5.5 MPa when properly cured, while Portland cement concretes typically have strengths in the 15 to 35 MPa range.[23]

Compressive strength data associated with the freeze/thaw programme are presented in Table 4-14. The strengths at comparable ages are very similar to those for ashes not subjected to the cyclic freeze/thaw process. In all cases, except Mix 1 after 5 cycles, moist curing substantially increased the unconfined compressive strength; for example, compare the strength of samples 31-33 with 43-45. Not only do the moist cured samples have better access to the water required for pozzolonic activity, but they are 7 days older than their non-moist-cured equivalents.

In Figures 4-16 and 4-17, the stress-strain behaviour of the four ashes over five and fifteen freeze/thaw cycles is shown graphically.

Table 4-12: Variation of Compressive Strength With Initial Moisture Content

Specimen No.	Sample Type	Moisture Content, %	Dry Unit Weight kN/m^3	Unconfined* Compressive Strength kPa
1	Mix 1, 1HE:2BH	19.9	13.05	217.9
2	Mix 1, 1HE:2BH	24.7	13.42	240.0
3	Mix 1, 1HE:2BH	29.7	13.76	108.1
4	Mix 1, 1HE:2BH	34.5	13.35	34.7
5	Mix 2, 2HE:1BH	19.9	14.04	287.0
6	Mix 2, 2HE:1BH	25.0	14.67	189.9
7	Mix 2, 2HE:1BH	30.2	14.26	38.1
8	Mix 2, 2HE:1BH	32.9	13.97	-
9	HE	18.0	12.06	90 **
10	HE	20.8	12.01	182 **
11	HE	23.7	12.01	183 **
12	HE	26.9	12.02	192 **
13	HE	30.0	12.07	269 **
14	HE	33.3	12.14	244 **
15	HE	36.1	12.08	194 **
16	HE	41.0	11.66	208 **
17	HE	43.9	11.42	132 **
18	BH	14.7	11.49	186.9
19	BH	19.9	11.87	177.5
20	BH	24.9	12.39	300.5
21	BH	30.7	12.83	221.0
22	BH	36.2	12.82	73.5
23	BH	42.1	12.18	-

* Maximum compressive stress divided by specimen area. No adjustments made for h/d ratio.

** From penetrometer; less reliable than uniaxial compression data

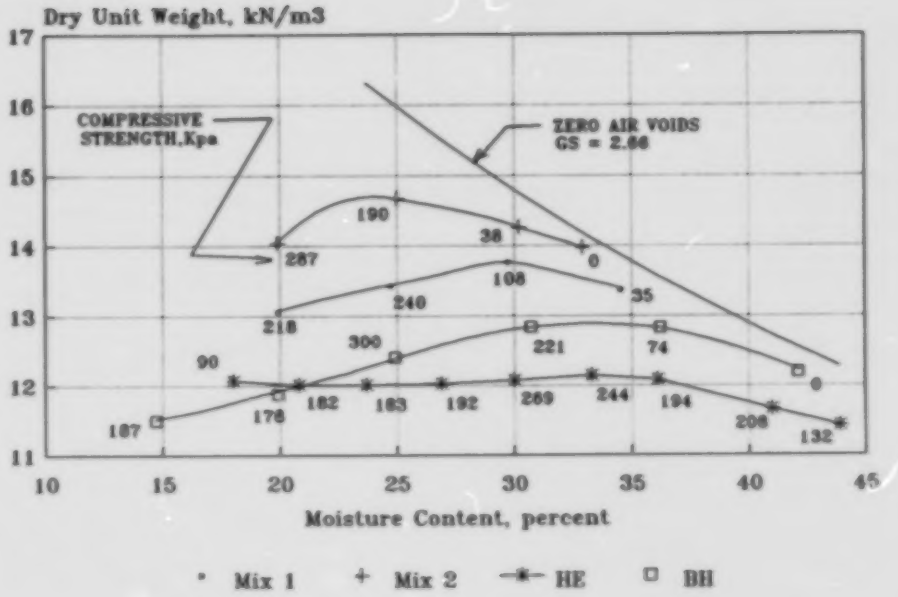

Figure 4-13: Unconfined Compressive Strength At Various Dry Unit Weights

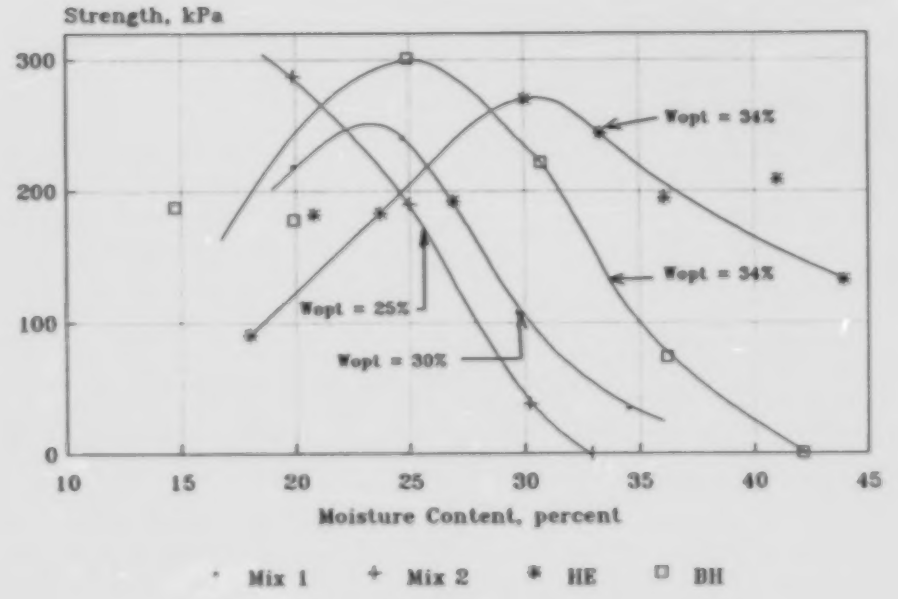

Figure 4-14: Unconfined Compressive Strength vs Moisture Content

Table 4-13: Variation of Compressive Strength With Specimen Age

Specimen No.	Sample Type	Age in Days	(2) Moisture Content, %	Dry Unit Weight, kN/m^3	Unconfined Comprehensive Strength, kPa	Average Qu kPa
(1)21,22	BH	0	33.4	12.82	147	
61		7	32.9	13.32	681	687
62		7		13.34	693	
63		14	32.7	13.37	800	757
64		14		13.37	714	
65		35	33.1	13.64	1160	
66		35		13.58	1416	1288
67		56	34.5	13.49	2563	2454
68		56		13.64	2346	
3	Mix 1, 1HE:2BH	0	29.7	13.76	108	
71		7	29.1	14.22	391	336
72		7		14.21	282	
73		14	27.7	14.36	589	560
74		14		14.49	531	
75		34	26.7	14.33	984	1014
76		34		14.56	1045	
77		55	29.8	14.58	1776	1692
78		55		14.46	1608	
6	Mix 2, 2HE:1BH	0	25.0	14.67	190	
81		7	26.0	15.00	316	317
82		7		15.06	319	
83		14	26.0	15.14	413	445
84		14		15.13	478	
85		34	25.2	15.27	1822	1685
86		34		15.29	1549	
87		55	25.3	15.41	3163	3094
88		55		15.35	3026	
14	HE	0	33.3	12.14	244(3)	
91		7	36.4	12.13	42	49
92		7		12.30	56	
93		14	35.5	12.44	130	144
94		14		12.46	158	
95		34	36.3	12.50	470	366
96		34		12.41	262	
97		55	35.2	12.52	542	536
98		55		12.60	530	

(1) Average of Samples 21 and 22; See Table 4-12
(2) At time of compression testing
(3) From Penetrometer; See Table 4-12

Table 4-14: Variation of Compressive Strength With Freeze/Thaw History

Specimen No.	Sample Type	No. Cycles	Age in Days	Unconfined Compressive Strength, kPa	Average Qu kPa
28*	Mix 1, 1HE:2BH	5	17	596	
29	Mix 1, 1HE:2BH	5	17	376	532
30	Mix 1, 1HE:2BH	5	17	624	
31	Mix 1, 1HE:2BH	15	37	1442	
32	Mix 1, 1HE:2BH	15	37	1044	1200
33	Mix 1, 1HE:2BH	15	37	1116	
34	Mix 2, 2HE:1BH	5	17	911	
35	Mix 2, 2HE:1BH	5	17	812	915
36	Mix 2, 2HE:1BH	5	17	1022	
37	Mix 2, 2HE:1BH	15	37	2496	
38	Mix 2, 2HE:1BH	15	37	1331	2009
39	Mix 2, 2HE:1BH	15	37	2198	
40	Mix 1, 1HE:2BH	5	10	475	
41	Mix 1, 1HE:2BH	5	10	469	554
42	Mix 1, 1HE:2BH	5	10	717	
43	Mix 1, 1HE:2BH	15	30	712	
44	Mix 1, 1HE:2BH	15	30	834	738
45	Mix 1, 1HE:2BH	15	30	668	
46	Mix 2, 2HE:1BH	5	10	442	
47	Mix 2, 2HE:1BH	5	10	365	486
48	Mix 2, 2HE:1BH	5	10	652	
49	Mix 2, 2HE:1BH	15	30	1414	
50	Mix 2, 2HE:1BH	15	30	768	1232
51	Mix 2, 2HE:1BH	15	30	1513	

* Specimens 28-39 were moist cured; 40-51 were not.

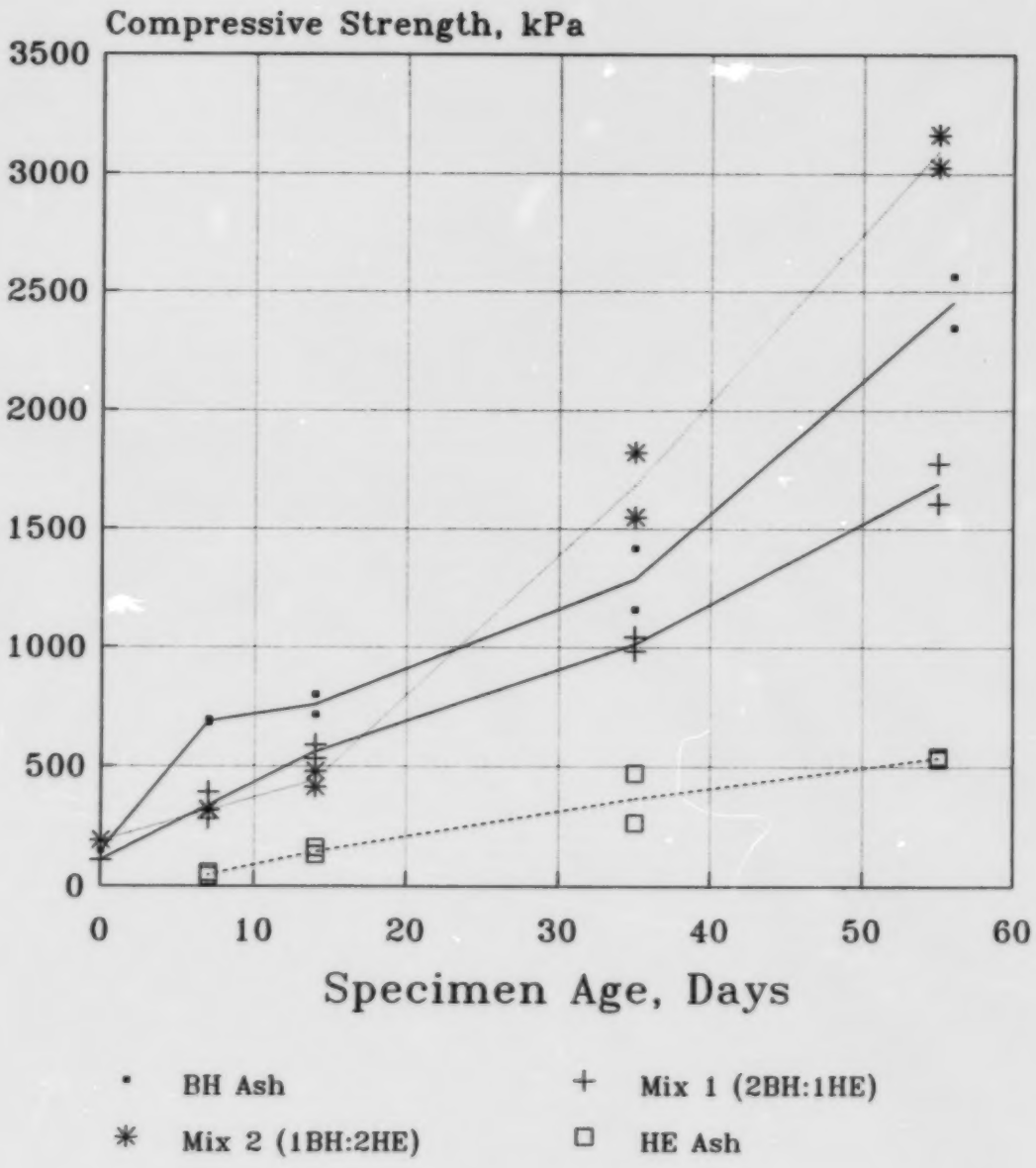

Figure 4-15: Compressive Strength vs Specimen Age

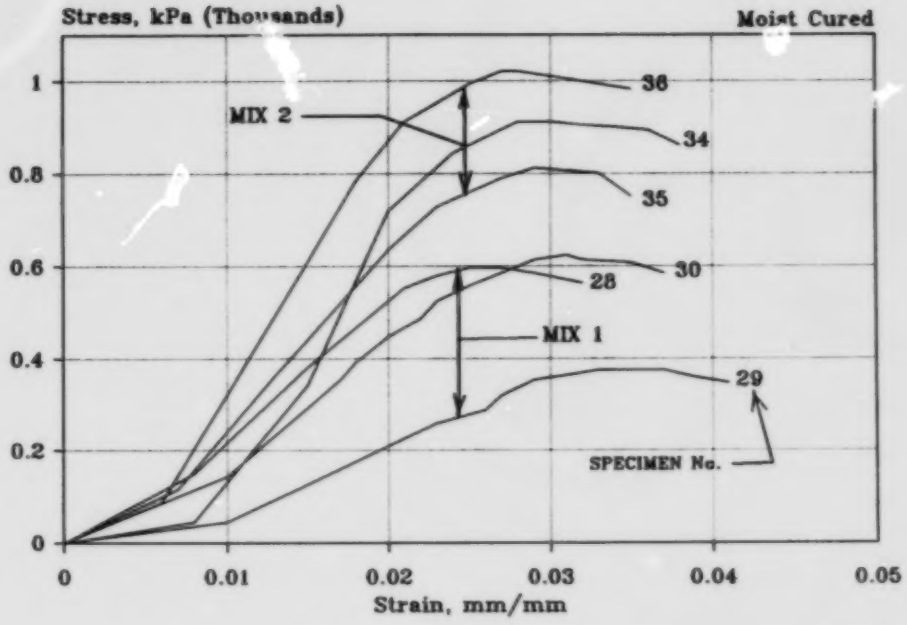

Figure 4-16: Stress-Strain Behaviour After 5 Freeze/Thaw Cycles

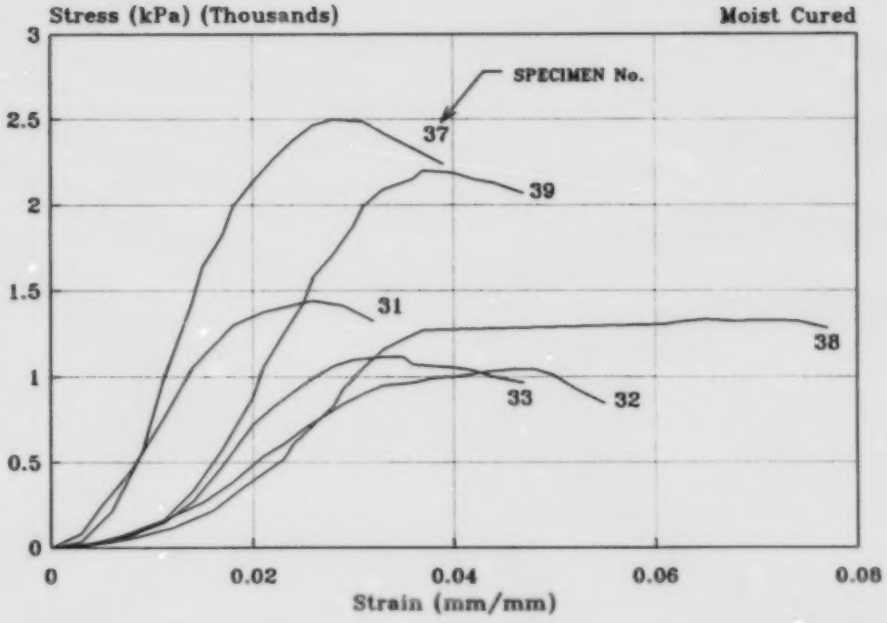

Figure 4-17: Stress-Strain Behaviour After 15 Freeze/Thaw Cycles

5.0 CONSIDERATION OF POTENTIAL CFBC WASTE UTILIZATION SCENARIOS

Circulating fluidized bed combustion of medium and high sulphur coals promises to be an efficient and cost effective way of utilizing this resource. Sulphur capture in the bed by limestone and/or oil shale sorbent is generally high and NO_x generation low and so environmental concerns are reduced. As a result of this, the use of CFBC technology by electrical utilities is expected to increase and so therefore is the quantity of waste. The cost of waste disposal is a major concern for the utility; the environmental consequences of disposal are of concern to both the utility and the public. In light of this, there has been pressure to develop economically attractive off-site uses for the ash.

In broad terms, the process of fluidized bed combustion generates two major solid wastes; the coarse bed discharge and the fine elutriated material or fly ash. Because the composition of these wastes can vary greatly, depending upon the nature of the coal burned, the type of sorbent employed in the bed and the specific operational characteristics of the CFBC unit, it is necessary to examine each ash product in detail before any recommendation for usage can be considered.

The particular operating regime which generated the ashes studied during the course of this project may not be the only one employed at the Chatham facility of N.B. Power. In addition, these tests, conducted without the input of additional sorbent in the form of limestone, may not represent the ultimate operational status of the unit. This being the case, it is only possible to describe potential uses for the ashes actually studied. Whether or not these scenarios could be extended to other ashes with different compositions is not known and would require further study at some future time.

From the results presented in Section 4.0 it is evident that the ashes generated by CFBC are different from those produced in conventional coal-fired generating plants (Section 3.0), and consequently may not be amenable to utilization in a similar manner. Information on past use of fly ash is useful in assessing the prospects for CFBC ash use.

According to Berry[16], only 27% of Canada's coal ash was utilized in 1977. Of the quantity that was put to use, 75.9% went to structural fill applications, 18.3% to Portland cement replacement, 0.3% as additions to Portland cement and 5.2% to all other projects (agriculture, fillers, ice control, mineral extraction, mineral wool production, grout, etc.). In 1980, a province-by-province breakdown of coal ash usage indicated variable utilization, ranging from 14.4% to 18.9%. These figures were generally dependant upon the level of construction activity in progress during the year.

5.1 Cementing Applications

The fly ashes derived from the combustion of lignite or sub-bituminous (Minto) coals are classified as Type F pozzolans if they have the following chemical properties[17]:

Table 5-1: Requirements For a Type F Pozzolan

Parameter	Class F Pozzolan
Silicon dioxide (SiO_2) + aluminum oxide (Al_2O_3) + iron oxide (Fe_2O_3), min, %	70.0
Sulphur trioxide (SO_3), max, %	5.0
Moisture content, max, %	3.0
Loss on ignition, max, %	6.0

Although the Minto coal fly ashes, derived from conventional utility boilers do meet the first of these criteria, they have generated little interest as pozzolanic admixtures in concrete because of their low Pozzolanic Activity Index (PAI) which is due to the high iron content; approximately 45%. From Table 3-5 it can be calculated that the level of SiO_2 + Al_2O_3 + Fe_2O_3 in these ashes is in the range of 85%-90%; well in excess of the minimum requirements for a Type F pozzolan. Nevertheless, as discussed in Section 3.3, these fly ashes can be utilized in certain specialized applications (mine back-fill for example), provided that the level of substitution is not too great.

The average percentage of SiO_2 + Al_2O3 + Fe_2O_3 in the Chatham CFBC ashes is closer to 65%; much lower than the conventional Minto coal fly ash but still within acceptable limits for a Type F Pozzolan, even though it is below the figure of 70.0 given in Table 5-1. The difference between these two ashes is that the iron oxide concentration in the CFBC ashes is much lower (6%-10%) thus improving its potential as a concrete admixture. As well, an examination of the chemical analysis presented in Table 4-4 will indicate that the level of SO_3 in these ashes is negligible. Assuming that the difference between total sulphur and SO_4 is attributable to SO_3, the resulting figures are all substantially below the 5.0% limit indicated in Table 5-1.

It has been suggested that the CaO levels in CFBC ashes should not exceed 7% if they are to be contemplated as aggregates in concrete[18]. The BH ash possesses the lowest concentration of CaO (8.75%), while the others range between 10% and 12%. Although these figures are higher than recommended, this alone would not rule out their use as concrete admixtures. The fact that they contain free lime is of concern. Free lime is prone to leaching which would leave voids in the concrete, allow the entry of water and lead ultimately to concrete failure. Nevertheless, an evaluation of the actual performance of these ashes in concrete would be required before any such decision could be made. The fact that the Chatham ashes are reasonably low in calcium (relative to CFBC ashes where limestone was used as a sorbent) and sulphates, and do demonstrate pozzolanic properties, renders them good candidates for further study, particularly in the context of mining usage. Additionally, it may be possible to enhance the pozzolanic/cementing properties of these ashes by fine grinding them. There is general agreement that the degree of pozzolanic activity, particularly with fly ashes, relates not only to their chemical compositions but also

to the grain size of the component particles.[16]

As a matter of interest, there is very limited potential for using high-calcium CFBC wastes in concrete. These wastes are usually not pozzolanic and they often contain elevated sulphate levels which makes them unsuitable for such use. Should the New Brunswick Electric Power Commission undertake further work using limestone as a sorbent in the CFBC, the resulting ashes will no doubt contain too much calcium for utilization in concrete.

Although the four CFBC ashes display some cementitious characteristics (because of their free lime content), the extent to which this might occur is expected to be low. The four ash types contain less than 0.8% free lime (Table 4-4).

5.2 Resource Recovery

The possibility of extracting magnetic iron concentrates from suitable fly ashes is one resource recovery scenario which has received consideration over the years. The process is simple, requiring nothing more than physical separation, and yields a product which has a number of potential uses. As described in Section 3.3, researchers at RPC were able to strip the iron component away from Minto coal fly ash and produce a material with a high specific gravity (3.9), which it was felt would be a good substitute for magnetite in coal washing circuits. Markets have also been identified for coal ash concentrates in the areas of iron ore supplementation and the production of iron salts for sewage coagulation[16].

Minto fly ash, derived from coal combustion in conventional power systems, possesses approximately 45% Fe_2O_3 (by weight) which even then makes the extraction of the iron fraction unattractive. The two CFBC parent ashes contain only 6.01% (HE) and 9.73% (BH) iron, concentrations too low to offer any economic opportunity.

The recovery of cenospheres from the ashes was also considered. It has been determined that the majority of the particles found in fly ashes which have been produced by conventional coal-fired generating plants are spherical in shape and range in diameter from 0.5 to 150 μm. On the other hand, the corresponding bottom ashes are usually comprised of larger, more irregular particles[16]. This is one aspect in which the ashes studied during this project differ from conventional fly ashes. The particle shape of all the Chatham CFBC ashes is irregular; electron microprobe and XRD examination identified insignificant quantities of spherical grains. The possibility of extracting cenospheres from these ashes is therefore unlikely. Traditional uses for cenospheres include low-cost substitutes for commercial glass microspheres, low-density fillers, and extenders in organic resins or Portland cement.

The levels of alumina and trace elements in the CFBC ashes are not sufficient to generate any interest in their recovery.

5.3 Production of Mineral Wool

The successful production of mineral wool depends upon achieving a suitable viscosity for the molten ash at the lowest possible temperature. Often, fluxing agents such as limestone and dolomite are employed to adjust the chemical composition of the melt and achieve the desired properties. The necessity of providing a flux may adversely affect the economics of the process. Recent work by the Coal Research Bureau at West Virginia University has shown that it is possible to produce a good quality mineral wool from raw, conventional coal ashes, therefore the previously established chemical criteria for mineral wool should be re-examined[16].

Whether or not the Chatham CFBC ashes would be suitable for mineral wool production is unknown. This could be determined with suitable test work.

5.4 Anti-stripping Additive Potential in Asphaltic Applications

Upon continued exposure to water, bituminous mixtures containing certain hydrophillic aggregates such as some quartzites and granites, lose shear strength. This occurs due to the replacement of asphalt cement films surrounding the aggregate particles with a film of water[19,20]. Aggregates such as (some) limestones and basalts which exhibit no such detrimental behaviour, are termed hydrophobic.

Potentially troublesome hydrophillic aggregates can be utilized to good effect by undergoing thorough drying prior to mixing and by addition of anti-stripping additives to the asphaltic concrete mixtures. Certain types of granitic aggregates in the Moncton area of New Brunswick which are hydrophillic, are used in combination with hydrated lime, as an anti-stripping additive[21]. Since the CFBC ashes under consideration here contain hydrated lime their potential for use as an anti-stripping agent may warrant consideration. Typical current usage of hydrated lime is about 5 kg per tonne of asphalt concrete[22].

5.5 Agricultural Uses of CFBC Ash

In agriculture, consideration has usually been given to the utilization of high calcium wastes as soil liming agents. Even in such applications, the lime equivalent of the residues may be as low as 33% that of calcium carbonate $CaCO_3$[18]. In many situations, unless the ash source is in close proximity to the point of intended use, transportation costs offset any benefit likely to be derived from their utilization in this manner.

The four CFBC ashes under consideration were produced without the use of limestone as a sorbent in the bed of the Chatham CFBC unit and consequently, they possess relatively low levels of calcium. It is not likely that agricultural liming would be a viable option in utilizing these ashes. To compound this disadvantage, the potential for heavy metals to leach from the ashes (Table 4-6) and be taken up and concentrated by various plant species, is a real concern. A high calcium CFBC waste is needed for liming and even then, plant growth studies would have to undertaken to assess the possible risk to the food chain.

5.6 Role as Neutralizing Agents

The neutralization capacities presented in Table 4-5 do not seem to indicate a high potential for the treatment of acid mine drainage or other acidic wastes. The best ash is the HE ash which has a capacity of only 219.67 kg of acid consumed per tonne of ash. When it is considered that commercial lime (95% CaO) possesses a neutralization capacity of approximately 1660 kg of acid (H_2SO_4) per tonne, and that the figure for calcium carbonate ($CaCO_3$) is roughly 965 kg of acid per tonne, the potential of the ash seems limited, unless the material were close to a market. As in the case of agricultural land treatment, if the source of the ash and the site of utilization are far removed from one another, transportation costs become a major factor. Again, if a different operating scenario were to be employed at the Chatham CFBC facility, and limestone were used as a sorbent, the potential use of the ash as a neutralizing agent would require re-examination.

The possibility may exist however, of back-hauling ash to Minto or Heath Steele Mines for incorporation into low hydraulic conductivity caps over abandoned acid-generating mine workings.

5.7 Use in Earthworks

As noted in Section 5.0, 27% of Canada's coal ash was used for construction purposes in 1977, three-quarters of which went to structural fill applications where it displaced higher value and/or heavier mineral aggregate alternatives. The remaining ash (73%) was presumably placed in landfills or stockpiled.

5.7.1 General Fill

Subject to environmental restrictions, the largest concern probably being the potential for contamination of groundwater by leached metals, the CFBC ashes could be placed as general fill using conventional earth moving equipment. The most attractive applications would be those where the site of such fill is desirable rather than tolerated, and where proximity to source or to a backhaul route would reduce the transportation costs. Old quarries, abandoned mines (See Section 5.6) and municipal landfills may all be candidates for such emplacement.

In addition to potential heavy metal leaching problems, other factors would have to be evaluated in the design of land reclamation schemes utilizing these ashes as general fill. The factors would include handling the ash at conditions other than the optimum moisture content, vulnerability to erosion and the potential for growing vegetative cover.

5.7.2 Structural Fill

Subject to the acceptability of the leachate chemistry, and the overall stability as determined by field trial, the ashes could be utilized as structural fill in highway construction. The HE

ash would be the easiest to handle with conventional earth moving equipment, as it is coarser in texture and more free-draining than the BH ash or mixtures containing it. Another advantage of the HE material is its relatively low dry unit weight, which at 12.2 kN/m^3 is lower than those of the other ash combinations tested. Low unit weights are advantageous where such fills are placed above compressible soils. In these situations, heavier conventional sand and gravel would result in increased foundation settlements and reduced overall fill heights for an equivalent factor of safety against bearing capacity failure.

Mix 2 ash, which comprises 2 parts HE ash to 1 part BH ash, is the most dense of the ash combinations (Figure 4-2). It also has the greatest unconfined compressive strength one or two months after compaction (Figure 4-15), and a relatively low hydraulic conductivity (Figure 4-7).

These characteristics might be utilized by applying the ash as a thin upper layer of an embankment where it would serve as a stabilizing cover and also reduce the downward water seepage to a more permeable zone.

5.7.3 Highway Surface Stabilization

Mix 2 ash or combinations of HE and BH ashes could be mixed with conventional aggregates, Portland cements, or bituminous cements or emulsions. Such mixtures could be tested for their applicability as surface treatments for unpaved roads or as stabilized bases beneath a bituminous wearing course.

Note that Mix 2, while exhibiting the highest unconfined compressive strength of the evaluated ashes, has only one quarter to one half the strength at 28 days of most conventional soil-cement mixtures[23].

5.7.4 Drainage and Filtration Applications

From a practical viewpoint, the baghouse (BH) ash and the two ash mixtures (Mix 1 and Mix 2) have similar hydraulic conductivities. In each case, the determined values are too high for the utilization of the ashes as lining materials for water retention structures, and too low to properly function in drainage or filter applications. The heat exchanger (HE) ash has a relatively high hydraulic conductivity and as such might be considered as a free draining layer in earthwork construction. The high solubility of heavy metals (As, Hg, and Pb) however would seriously restrict its use for such purpose.

6.0 CONCLUSIONS AND DISCUSSION

6.1 Chemical Composition

Table 6-1 presents a comparison of the major chemical properties of Minto Coal, Albert oil shale and the ashes resulting from their combustion, either in conventional power boilers or in the Chatham circulating fluidized bed combustion unit. The figures for Minto coal are those given in Table 3-3: Semi-quantitative ICP Multi-Element Analysis Of Minto Coal. In that table three sets of figures were listed, one for Coal Creek coal and two for Dragline 8200 coal. Table 6-1 presents an average of these three data sets. Although the Dragline 8200 coal was the predominant coal used in the generation of the CFBC ashes under study in this project (70%), the figures in Table 6-1 have not been weighted to reflect this ratio. The Albert oil shale figures come from Table 3-8, the Minto coal ash figures from Table 3-5 (average of six sets of figures), the figures for Albert oil shale ash from Table 3-10 (average of four sets of data) and the Chatham CFBC ash figures from Table 4-4 (average of four sets of data).

Minto coal ash possesses, on average, 30.85% SiO_2, while Albert oil shale contains 49.52%. Since the ratio of oil shale to coal employed in the Chatham test burns which generated the ashes studied during this project was 2:1 (Section 3.1), an SiO_2 level of perhaps 43% might be anticipated. In fact, the average percentage of SiO_2 found in the CFBC ashes is 51.05%. Some of this discrepancy may be accounted for by the fact that the figures reported in Table 6-1 (apart from those for the CFBC ashes) are average figures and that they can vary by as much as ± 10%. For example, Leveille[11h] recorded an SiO_2 level of 54.29% for one of two Albert Mines oil shale ash samples considered during that study.

The coal ashes resulting from conventional combustion are much higher in Al_2O_3 (12.25%) than the CFBC ashes (6.95%), while the oil shale ash contains even higher levels; 21.52%. Based on the 2:1 oil shale/coal ratio, an Al_2O_3 figure for the CFBC ashes closer to 18% might be expected. Again, given the range of values possible for Minto coal/Albert oil shale ashes and the fact that perhaps one half of the insoluble material occluded by the silica (Section 4.4) could be alumina, the levels of Al_2O_3 in the CFBC ashes are likely to be significantly higher than here reported.

As described previously in Section 3.3, Minto coal ash is well known for its high iron oxide content. This concentration (44.98%) is significantly higher than the figures of 7.95% and 7.67% reported in Table 6-1 for Albert oil shale ash and Chatham CFBC ash. A calculation based on the 2:1 oil shale/coal ratio indicates that a figure for the CFBC ashes closer to 20% would be more likely. In this case as well, accounting for the range of values attributable to coal/oil shale ashes and the allowance for the presence of insolubles, a more reasonable figure of 15% is possible. Note also that the Minto coal bottom ash contained 36.58% Fe_2O_3 and the fly ash 45.31% (Table 3-5), while the HE ash (coarser) contained 6.01% and the BH ash (finer) contained 9.73% (Table 4-4). In both cases, the finer ashes revealed greater concentrations of iron oxide.

Table 6-1: Comparison of the Chemical Compositions of Coal/Oil Shale Ash (Wt.%)

Constituent	Minto Coal	Albert Oil Shale	Minto Coal Ash	Albert Oil Shale Ash	Chatham CFBC Ashes
SiO_2	5.18	40.78	30.85	49.52	51.05
Al_2O_3	1.90	12.87	12.25	21.52	6.95
Fe_2O_3	8.26	5.20	44.98	7.95	7.67
Mn_3O_4	---	0.12*	---	---	0.12*
MgO	0.17	3.58	0.33	5.95	5.58
CaO	0.33	10.75	5.00	9.86	10.43
K_2O	0.91	1.61	0.62	2.61	0.87
Na_2O	0.26	1.28	0.37	2.59	0.45
P_2O_5	0.16	<0.01	1.32	---	0.39
TiO_2	0.18	0.37	0.58	---	0.14
SO_3	0.58	---	1.49	---	---
SrO	---	---	---	---	---
BaO	0.01	---	0.32	---	---
Fusion Loss	0.13	21.62**	---	---	---

* MnO
** Loss on Ignition

The average percentage of MgO in Minto coal ash is 0.33 while the average percentage in the oil shale ash is 5.95. The average percentage of MgO in the CFBC ashes is 5.58% which, given the 2:1 oil shale-to-coal ratio used in the Chatham CFBC unit, appears to be realistic.

The CaO content of the CFBC ashes is 10.43%, which is consistent with the values given for Minto coal ash and Albert oil shale ash.

The minor element chemistry of Minto coal, Albert oil shale , Minto coal ash, Albert oil shale ash, and the CFBC ashes are summarized in Table 6-2. These figures are obtained from the Tables in Section 3.0 and are averages (where applicable) of the values reported there. Trace element concentrations in the Chatham CFBC ashes are, in most cases, similar to those in the parent coal and oil shale and consequently are not high enough to be economically encouraging.

In summary, it can be stated that the combustion of Minto coal with Albert oil shale in a circulating fluidized bed combustion system does not produce any concentration of minerals, major oxides or trace elements which greatly exceed those found in either the parent materials or their ashes.

Table 6-2: Comparison of Trace Elements of Coal/Oil Shale Ash

Sample	Minto Coal (ppm)	Albert Oil Shale (ppm)	Minto Coal Ash (ppm)	Albert Oil Shale Ash (ppm)	Chatham CFBC Ashes (ppm)
As	145	–	18	–	925
Mn	646	–	400	–	900
Zn	253	84	16	–	100
Pb	116	54	18	–	150
Cd	0	–	<1	–	<10
Ni	18	32	12	40	55
Ba	9	102	<1	–	<0.001
Co	72	–	18	–	–
Cu	0	35	3	80	70
Li	22	67	8	–	<500
Cr	0	40	5	–	90
Rb	–	41	<8	–	<50
Sr	–	943	60	–	<500
V	–	25	128	260	170
Ag	–	3.3	<0.5	–	<0.1

6.2 Leachate Considerations

Table 6-3 presents a comparison of the sum of the concentrations of the leached ionic species and the TDS (total dissolved solids) obtained from the Equilibrium Leach Tests described in Section 4.5.

In each case the sum of the ions is less than the TDS values; by 31% on average and with a range of 12% to 48%. It will also be noted that the differences are greater for the ash mixes (Tests 5-8) than for the parent HE and BH ashes (Tests 1-4).

There are likely other ionic species present in the leachates analyzed, notably soluble silica, $Ca(OH)_2$ from the free lime present, chlorides from the hydrochloric acid used to perform the leaching and perhaps aluminum, iron, magnesium, and carbonate ions. These additional ionic species undoubtedly account for the difference between the ion sums and TDS figures. The obvious decrease in concentration of the recorded ions in the ashes when the BH and

Table 6-3: Comparison of Concentrations of Leached Ionic Species and TDS

Test No.	Ion Sums (mg/L)	TDS (mg/L)	% Difference (TDS as Reference)
1	3103	3544	-12
2	2978	3561	-16
3	2359	2829	-17
4	2166	2935	-26
5	1657	2982	-44
6	1840	2919	-37
7	1602	3078	-48
8	1652	3069	-46

HE ashes are mixed to form Mix 1 and Mix 2 is not readily explainable. It seems likely however that chemical reactions are taking place between or among species in the two ashes which give rise to precipitates (i.e. insoluble minerals) thereby decreasing the concentration of the recorded ions. At the same time the concentration of unrecorded ionic species must be increasing to retain the TDS values approximately constant.

As discussed previously, the tendancy for heavy metals (As, Pb and Hg) to readily leach from the Chatham CFBC ashes presents a significant point of concern when assessing various utilization scenarios. The Guidelines for Canadian Drinking Water Quality[24] suggest the following levels for these metals:

As	0.05 mg/L (currently under review)
Pb	0.01 mg/L (proposed)
Hg	0.001 mg/L (adopted)

Examination of Table 4-10 indicates that the leachates from all four ashes contain greater concentrations than these and are therefore potential sources of groundwater contamination.

6.3 Physical and Mechanical Properties

The parent ashes and ash mixtures were subjected to routine moisture/density relationship tests for evaluation of their behaviour as compacted fill. Each ash proved to be relatively light-weight, with maximum dry unit weights of 80 to 100 percent of those of typical loose uniform sands. The mineral specific gravities, Gs, were 2.66 and 2.67 for BH and HE ash respectively, consistent with the ash mineralogy. Quartz, which is the major component, for example, (See Table 4-2) has a specific gravity of 2.65. The fact that compacted densities are lower than those for natural sands with similar primary minerals is presumably a

reflection of differences in grain shape. The ashes do not, however, comprise large proportions of well-rounded particles (Section 4.3).

The hydraulic conductivity for compacted molds of BH ash and both mixtures comprising BH ash are very similar and comparable to conductivities for fine silty sands, but 3 to 30 times lower than might have been anticipated for natural sands of similar gradation. Compacted HE ash is 60 to 90 times more permeable than the other ashes, and although still lower, has a hydraulic conductivity more closely comparable to that anticipated for a sand of similar gradation. Significant variations of hydraulic conductivity were observed in the course of each test, the cause or causes of which is not fully understood. Potential mechanisms for these variations include changes in the degree of sample saturation and concurrent processes of mineral dissolution and/or precipitation at grain contacts.

Although each of the hydraulic conductivity specimens lost a significant amount of weight during passage of leachate due to mineral dissolution, the cementing process at particle contacts resulted in substantial strength gain with curing time. Two months after molding, the unconfined compressive strength of the compacted HE ashes was approximately 500 kPa while the BH and ash mixtures were 3 to 6 times stronger at this age. Interestingly, Mix 2 which comprised one part BH to 2 parts HE, was consistently the strongest of the ashes after an age of one month.

The freeze/thaw testing was quantitatively somewhat inconclusive. The test method followed the ASTM D 560-82 procedure for soil-cement mixtures but with the brushing stage omitted because of sample fragility. Most samples lost weight during the testing period but this probably has little practical significance because field conditions would be quite different from the controlled ones of the test. Qualitatively the freeze/thaw tests indicate that when properly compacted these ashes maintain their integrity when subjected to repeated freeze/thaw cycles. This does not necessarily mean that these materials would not be susceptible to disintegration and/or frost heaving given access to water during freezing - a distinct possibility which would have to be evaluated before their utilization as fills in critical areas. Field behaviour in a freeze/thaw environment would depend on access to water and on the rate and history of temperature change.

Freeze/thaw activity appears to have little effect on compressive strength; the strength of freeze/thaw specimens is similar to that of regularly cured specimens of comparable age.

Supplementary testing of specific physical and mechanical properties of these ashes would be required before their utilization on a large scale. Such testing would have to be designed to match actual applications and field conditions.

6.4 Utilization Scenarios

Of the numerous potential utilization scenarios presented in Section 5.0, there appear to be only a few which look immediately promising. Cementing applications might be considered, even though a comparison of the specifications for a Type F pozzolan and the chemical properties of the CFBC ashes (Section 5.1) do not appear to support this use. Testing of

various CFBC ash cements could be worthwhile, particularly if fine grinding were to be investigated in conjunction with this work.

Due to the presence of free lime in these ashes, there may be some potential for their use as anti-stripping agents in asphaltic applications. Obviously though, even if the ash properties were suitable for such usage, the cost and availability of current substitutes, plus the cost of transportation would have to be closely examined in order to determine viability.

Although use of the Chatham CFBC ashes as neutralizing agents for acid mine drainage control does not seem promising, there may be an opportunity to incorporate the ash into low hydraulic conductivity caps over abandoned acid-generating mine workings.

Use as fills, either general or structural, looks promising when only the physical properties of the CFBC ashes are taken into account. In all cases though, the generation and control of undesirable leachates would have to be considered. Given the increasing attention being directed to the problem of ground water contamination in New Brunswick, such usages could present a major challenge.

Resource recovery from the ashes, using the ash as agricultural liming materials, or employing them for filtration and drainage purposes are all either economically unattractive, or undesirable due to the possibility of heavy-metal leaching.

7.0 RECOMMENDATIONS

The tendency for heavy metals to leach from the CFBC ashes will impact adversely on all the favourable potential utilization scenarios discussed in Section 5.0.

Although laboratory leaching tests generate useful information regarding the behaviour of CFBC ashes, the data cannot be directly applied to actual field conditions. It would therefore be useful to conduct long-term leaching tests which would more closely represent true environmental conditions. The resulting information would be helpful in more fully assessing the proposed utilization scenarios. It would also provide data on the consequences of long-term storage and/or disposal of the ashes.

One of the more likely uses for the CFBC ashes is for highway surface stabilization as described in Section 5.7.3. In order to further investigate this idea, a short section of highway could be constructed, near Chatham for example, where it would be assessed over an extended period of time. The study would examine the durability of the surface, and at the same time monitor the generation of leachates.

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APPENDICES

A. Terms of Reference
B. Raw Data From XRD Tests
C. Equilibrium Leach Test (ELT) Procedure
D. ASTM Standard D 698-78 Method A "Test Methods for Moisture-Density Relations of Soils and Soil-Aggregate Mixtures, Using 5.5-lb (2.49-kg) Rammer and 12-in. (304.8-mm) Drop"
E. ASTM Standard D 560-82 "Methods for Freezing-and-Thawing Tests of Compacted Soil-Cement Mixtures"
F. ASTM Standard D 1633-84 "Compressive Strength of Molded Soil Cement Cylinders"

APPENDIX A
Terms of Reference

INFORMATION TO BIDDERS RR:
PROPOSALS FOR OIL SHALE/COAL ASH
CHARACTERIZATION AND POTENTIAL UTILIZATION

OBJECTIVES:

To undertake for the New Brunswick Department of Natural Resources and Energy, a series of laboratory-scale chemical, physical and geotechnical characterization tests on oil shale and coal ash from the New Brunswick Electric Power Commission demonstration combustor in Chatham, New Brunswick. The tests will be used to determine if potential by-products are recoverable, and if the ash can be otherwise utilized. Several hundred tonnes of oil shale/coal ash will be produced from the Chatham combustor trials. If the trials are successful and the decision is made to proceed with a commercial plant, hundreds of thousands of tonnes of ash will become available. The viable utilization of this ash could provide an additional incentive for the development of New Brunswick's oil shale resources.

WORK TO BE PERFORMED:

Without in any way limiting the scope of the work, the study should contain the following elements as minimum requirements:

1. To review the pertinent literature on the physical, mineralogical and chemical characteristics of New Brunswick's Albert Formation oil shale and oil shale ash and the physical, mineralogical and chemical characteristics of New Brunswick's Minto coal and coal ash.

2. To review the pertinent literature on the physical, mineralogical and chemical characteristics of the New Brunswick Havelock or Elmtree limestones used as a partial sorbent in the combustor trials.

3. To review the nonproprietary literature on the operation and particularly the ash discharge system operation of the N.B. Power, Chatham circulating fluidized bed combustor.

4. To receive at no charge to you the relative weight percentages of the feeds of oil shale, coal and limestone utilized in producing the ash for this study.

5. To receive at your chosen location, at no expense to you, approximately 250 kg of ash cooler ash from the fluidized bed heat exchanger (FBHE) and approximately 250 kg of baghouse ash from the Chatham combustor. The ash will tentatively be delivered in four (4) 205 litre metal drums.

6. To prevent, by all means possible, the ash from contacting moisture, except where otherwise directed.

7. To mix or otherwise thoroughly homogenize the 250 kg sample of fluidized bed heat exchanger (FBHE) ash.

8. To mix or otherwise thoroughly homogenize the 250 kg of baghouse ash.

9. To carry out a size analysis on a representative sample of each of the FBHE ash and baghouse ash using screens with the following Tyler Mesh (M) sizes: 6M, 10M, 20M, 60M, 100M, 200M, 270M, 325M, 400M, minus 400M (Pan). If greater than 50 weight per cent passes the 200M screen, the fraction that passes the 200M screen shall be size analyzed by hydrometer analysis.

10. To plot size distribution graphs for the FBHE ash and baghouse ash.

11. To select representative samples of the FBHE ash and baghouse ash and to carry out the following determinations and analyses:

    a. Relative percentage of minerals.
    b. Weight percentage of the oxides: SiO_2, Al_2O_3, $FeO*Fe_2O_3$, CaO, MgO, Na_2O, K_2O, P_2O_5, TiO_2.
    c. Free lime (CaO) content.
    d. Total organic carbon content.
    e. Quantities of the following metals and trace elements: Cu, Pb, Zn, Ag, Li, Ba, V, Cr, Cd, Ni, Mn, Hg, As, Sr, Rb, and U. Elements that can be, shall be determined by Inductively Coupled Plasma Analyses, otherwise they shall be determined by Atomic Absorption Spectrometry.
    f. Total content of phosphates (PO_4^{-3}) and nitrates (NO_3^{-1}).

12. To select a representative sample of each of the FBHE ash and baghouse ash and carry out leaching tests on each using (a) deionized water and (b) hydrochloric acid leach with a pH of 4.5.

13. To test the leachates generated in Section 12a and 12b for:

    a. pH
    b. Total dissolved solids (TDS)
    c. Na, K, Ca, Pb, Hg, As, SO_4^{-2}, PO_4^{-3}, and NO_3^{-1} contents.

14. To select a representative sample of each of the FBHE ash and baghouse ash and to form a 4 inch (101.6 mm) mold from each for the purpose of determining moisture-density relations in accordance with ASTM-D698-78. Deionized water should be used for mold formations.

15. To determine the optimum moisture content and maximum density on each of the molds described in Section 14 above.

16. To carry out on each of the molds on the day of formation, a permeability test with deionized water and retain the first volume of pore fluid for testing as per Section 19 below.

17. To seal and store each of the molds at room temperature (22°) in a humid environment (100% humidity).

18. To carry out on each of the molds after 40 days in storage, a permeability test using a hydrochloric acidic leach with a pH of 4.5.

19. To recover the first volume of pore fluid from the permeability tests described above in Sections 16 and 18 and from each of the fluids determine:

    a. pH
    b. Total dissolved solids (TDS)
    c. Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents.

20. To carry out, following the tests in Sections 18 and 19, a uniaxial unconfined compression test on each of the molds described above in Sections 14 through 19.

21. To tabulate for the molds of FBHE ash and the baghouse ash the uniaxial strength, the change over 40 days of permeability, leachate pH, leachate TDS, and leachate Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents for both deionized water and acidic leach conditions.

22. To mix and thoroughly homogenize FBHE ash and baghouse ash in the ratio of 2 parts baghouse ash by weight to 1 part FBHE ash by weight. For the purpose of description this mixed ash will here be called "Mixed Ash 1".

23. To prepare a sufficient quantity of Mixed Ash 1 to conduct the tests and determinations described in Sections 24 through 44 below.

24. To carry out on a representative sample of Mixed Ash 1 described above in Section 23 a size analysis by combined screen and hydrometer analysis and to plot a size distribution graph.

25. To select representative samples of Mixed Ash 1 and to carry out the following determinations and analyses:

    a. Relative percentage of minerals.
    b. Weight percentage of the oxides: SiO_2, Al_2O_3, $FeO*Fe_2O_3$, CaO, MgO, Na_2O, K_2O, P_2O_5, TiO_2.
    c. Free lime (CaO) content.
    d. Total organic carbon content.
    e. Quantities of the following metals and trace elements: Cu, Pb, Zn, Ag, Li, Ba, V, Cr, Cd, Ni, Mn, Hg, As, Sr, Rb, and U. Elements that can be, shall be determined by Inductively Coupled Plasma Analyses, otherwise they shall be determined by Atomic Absorption Spectrometry.

26. To select a representative sample of Mixed Ash 1 and carry out leaching tests on each using (a) deionized water and (b) hydrochloric acid leach with a pH of 4.5.

27. To test the leachates generated in Section 26a and 26b for:

    a. pH
    b. Total dissolved solids (TDS)
    c. Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents.

28. To select six (6) representative samples of Mixed Ash 1 and to form six (6) 4 inch (101.6 mm) molds for - in addition to other tests - the purpose of determining moisture-density relations in accordance with ASTM-D698-78. Deionized water should be used for mold formations.

29. To determine the optimum moisture content and maximum density on each of the molds described in Section 28 above.

30. To carry out on one (1) of the molds described above in Sections 28 and 29, on the day of formation, a permeability test with deionized water and to retain the first volume of pore fluid for testing as per Section 32 below.

31. To carry out on one of the molds described above in Sections 28 and 29, on the day of formation, a permeability test with hydrochloric acidic leach with a pH of 4.5 and to retain the first volume of pore fluid for testing as per Section 32 below.

32. To test the leachates generated in Sections 30 and 31 for:

    a. pH
    b. Total dissolved solids (TDS)
    c. Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents.

33. To carry out on the day of formation, a uniaxial unconfined compression test on the two (2) molds described above in Sections 30 and 31.

34. To, immediately following formation, seal and store at room temperature (22°C) and in a humid environment (100% humidity) two (2) of the molds described above in Section 28.

35. To retrieve, after ten (10) days in storage, one (1) of the molds described above in Section 34, and to carry out a permeability test with hydrochloric acidic leach with a ph of 4.5 and to retain the first volume of pore fluid for testing as per Section 36 below.

36. To test the leachates generated in Section 35 for:

    a. pH
    b. Total dissolved solids (TDS)
    c. Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents.

37. To carry out a uniaxial unconfined compression test on the mold described above in Section 35.

38. To retrieve, after forty (40) days in storage, the remaining mold described above in Section 34, and to carry out on it the same permeability, leachate, and compression tests described in Sections 35, 36, and 37 above.

39. To carry out 40 days after mold formation freezing and thawing tests on the remaining two (2) molds described above in Section 28. The freezing and thawing tests can be effected by modifying Method A of ASTM D560-82. The essential modification required is that cement must not be added as per Section 5.3.5 of the ASTM test.

40. To run one (1) of the molds described above in Section 39 through five (5) cycles of freezing and thawing, a cycle being as described in Section 5.3.5 of ASTM D560-82.

41. To run one (1) of the molds described above in Section 39, not the one described in Section 40, through fifteen (15) cycles of freezing and thawing.

42. To carry out, following the freezing and thawing tests, a uniaxial unconfined compression test on the two (2) molds described above in Sections 39, 40, and 41.

43. To tabulate and graph (where relevant) for Mixed Ash 1, the rate of change over forty (40) days of permeability, leachate pH, leachate TDS, and leachate Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents.

44. To tabulate and graph for Mixed Ash 1, the rate of change of uniaxial strength and the results of the freezing and thawing cycles.

45. To mix and thoroughly homogenize FBHE ash and baghouse ash in the ratio of 1 part baghouse ash by weight to 2 parts FBHE ash by weight. For the purpose of description, this mixed ash will here be called "Mixed Ash 2".

46. To prepare a sufficient quantity of Mixed Ash 2 to conduct the tests and determinations described in Sections 47 through 63 below.

47. To carry out on a representative sample of Mixed Ash 2 described above in Section 46 a size analysis by combined screen and hydrometer analysis and to plot a size distribution graph.

48. To select representative samples of Mixed Ash 2 and to carry out the following determinations and analysis:

    a. Relative percentage of minerals.
    b. Weight percentage of the oxides: SiO_2, Al_2O_3, $FeO*Fe_2O_3$, CaO, MgO, Na_2O, K_2O, P_2O_5, TiO_2.
    c. Free lime (CaO) content.
    d. Total organic carbon content.

e. Quantities of the following metals and trace elements: Cu, Pb, Zn, Ag, Li, Ba, V, Cr, Cd, Ni, Mn, Hg, As, Sr, Rb, and U. Elements that can be, shall be determined by Inductively Coupled Plasma Analyses, otherwise they shall be determined by Atomic Absorption Spectrometry.

49. To select a representative sample of Mixed Ash 2 and carry out leaching tests on each using (a) deionized water and (b) hydrochloric acid leach with a pH of 4.5.

50. To test the leachates generated in Section 49a and 49b for:

    a. pH
    b. Total dissolved solids (TDS)
    c. Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents.

51. To select four (4) representative samples of Mixed Ash 2 and to form four (4) 4 inch (101.6 mm) molds for - in addition to other tests - the purpose of determining moisture-density relations in accordance with ASTM-D698-78. Deionized water should be used for mold formations.

52. To determine the optimum moisture content and maximum density on each of the molds described in Section 51 above.

53. To carry out on one (1) of the molds described above in Sections 51 and 52, on the day of formation, a permeability test with deionized water and to retain the first volume of pore fluid for testing as per Section 54 below.

54. To test the leachates generated in Section 53 for:

    a. pH
    b. Total dissolved solids (TDS)
    c. Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents.

55. To carry out on the day of formation, a uniaxial unconfined compression test on the mold described above in Section 53.

56. To, immediately following formation, seal and store at room temperature (22°C) and in a humid environment (100% humidity) one (1) of the molds described above in Section 51.

57. To retrieve, after ten (10) days in storage, the mold described above in Section 56, and to carry out a permeability test with hydrochloric acidic leach with a pH of 4.5 and to retain the first volume of pore fluid for testing as per Section 58 below.

58. To test the leachates generated in Section 57 for:

    a. pH
    b. Total dissolved solids (TDS)
    c. Na, K, Ca, Pb, Hg, As, and SO_4^{-2} contents.

59. To carry out a uniaxial unconfined compression test on the mold described above in Section 57.

60. To carry out freezing and thawing tests on the remaining two (2) molds described above in Section 51. The freezing and thawing tests can be effected by modifying Method A of ASTM D560-82. The essential modification required is that cement must not be added as per Section 5.3.5 of the ASTM test.

61. To run one (1) of the molds described above in Section 60 through five (5) cycles of freezing and thawing, a cycle being as described in Section 5.3.5 of ASTM D560-82.

62. To run one (1) of the molds described above in Section 60, not the one described in Section 61, through fifteen (15) cycles of freezing and thawing.

63. To carry out, following the freezing and thawing tests, a uniaxial unconfined compression test on the two (2) molds described above in Sections 60, 61, and 62.

64. To tabulate and graph (where relevant) for Mixed Ash 2, the permeability, leachate chemistry, uniaxial strength and freezing and thawing data.

65. To label and retain all ash samples, subsamples, and powders, all leachate and permeability test fluids, and all molds for a period of one (1) year following submission of the final report.

66. To determine from an assessment of the results of this study and from a review of the relevant literature whether there are any potentially recoverable products from the different ash types.

67. To determine from an assessment of the results of this study and from a review of the relevant literature (and from other local industry and government sources) whether the ash could be used for soil stabilization, as a filler in asphaltic concrete, as a source of lime for waste neutralization or agriculture. It is required that the ash be evaluated for its potential in land reclamation, as a sanitary landfill and whether it has any nutrient values.

68. To prepare for editing five (5) double-spaced typed copies of a draft report to be submitted to the Director of the Energy Branch, Department of Natural Resources and Energy, P.O. Box 6000, Fredericton, New Brunswick, E3B 5H1, on or before the 12th day of May, 1989.

69. To insure that the draft report shall contain, but not be limited to:

    a. An abstract (bilingual), table of contents, and an introduction.
    b. A background description of the relevant data on the Albert oil shale, the Minto coal, and the Elmtree or Havelock limestone.

c. A summary description of the Chatham circulating fluidized bed combustor with particular emphasis on the ash discharge system.
d. A complete tabular, graphic and descriptive account of all the laboratory methodologies, and analytical and geotechnical data resulting from this study.
e. A description of the potential by-products recoverable from the ash and whether the ash has any potential utility in the construction, waste management, or agricultural sectors as more fully described above.
f. Recommendations for follow-up studies.

70. To receive and correct the edited draft report.

71. To prepare an unbound original copy and ten (10) single- spaced typed copies of a final report to be submitted to the Director of the Energy Branch, Department of Natural Resources and Energy, P.O. Box 6000, Fredericton, New Brunswick, E3B 5H1, on or before the 28th day of July 1989.

72. To insure that the final report is free of spelling, grammatical, syntactical, typographical, and mechanical errors and is in all other ways ready for publication, if it were so decided.

73. To provide with the final report a diskette if the report were typed on a word processor.

INFORMATION TO BIDDERS:

1. The Energy Branch of the New Brunswick Department of Natural Resources and Energy will deliver to the successful tenderer at the Department's expense four (4) drums of ash weighing approximately 500 kg.

2. The Energy Branch will provide copies of any public information published by the New Brunswick Department of Natural Resources and Energy considered relevant to this study at no cost to the successful bidder.

3. The Energy Branch will appoint a staff geologist to represent the Department of Natural Resources and Energy and to communicate with the successful tenderer.

4. The Energy Branch shall retain ten (10) per cent of the tendered amount until submission and approval of the final report.

5. The successful bidder shall enter into a formal contract with the New Brunswick Department of Natural Resources and Energy wherein full details of the work and the expected project completion date will be given.

6. Bidders will not be reimbursed for preparing their proposal. The lowest or any bid may not be accepted.

TENDER REQUIREMENTS:

In submitting a proposal to tender, bidders should provide the following:

1. A description of the methods by which the work to be performed will be achieved.

2. A time table detailing when the various aspects of the work shall be completed, bearing in mind that the project shall begin when the successful tender is announced and that seven (7) months is deemed a reasonable time to complete the project.

3. The names, qualifications and experience of all personnel (including subcontractors and their personnel) to be used on the project, and the amount of time each of these personnel will give to the work.

4. The fees, charges, and estimates of expenses and the total estimated cost of the project. The following should be included as necessary minimum requirements:

   a. The hourly or per diem rate of all personnel assigned to the project.
   b. Various sample preparation unit costs.
   c. A complete listing of all analytical unit costs.
   d. An estimate of the material costs to prepare the final unbound report.
   e. An estimate of the cost of the ten (10) bound copies of the final report.
   f. The total estimated cost of the project.

5. An indication of how frequently interim payments shall be required.

6. An assurance that monthly interim, typed progress reports shall be submitted to the Department staff geologist monitoring the project. Reports shall be provided on the final working day of the month and include a summary of work completed, status of the work, problems encountered and methods employed to resolve them, and an indication of the work planned for the following month.

7. An assurance that verbal progress reports shall be provided upon request of the Department of Natural Resources and Energy.

8. Three (3) copies of the proposal should be submitted on or before 16:30 hours A.D.S.T. on the 2nd day of December, 1988 to the attention of Mr. C. St. Peter, Department of Natural Resources and Energy, Energy Branch, Room 150, Hugh John Flemming Forestry Centre, R.R. #10, Regent Street Extension, Fredericton, New Brunswick, E3B 6H6.

SUMMARY OF TESTS FOR OIL SHALE/COAL ASH UTILIZATION

TEST DESCRITPION	FBHE*	BH+	MIXED ASH 1	MIXED ASH 2
Size analysis	1=	1	1	1
Mineralogy	1	1	1	1
Major oxides	1	1	1	1
Free CaO	1	1	1	1
Total Organic Carbon (TOC)	1	1	1	1
Trace chemistry	1	1	1	1
Ash leachate, pH = 7	1	1	1	1
Ash leachate, pH = 4.5	1	1	1	1
Mold formation	1	1	6	4
Optimum moisture/maximum density	1	1	6	4
Permeability, pH = 7, day 1	1	1	1	1
Permeability, pH = 4.5, day 1	0	0	1	0
Permeability, pH = 4.5, day 10	0	0	1	0
Permeability, pH = 4.5, day 40	1	1	1	1
Mold leachate, pH = 7, day 1	1	1	1	1
Mold leachate, pH = 4.5, day 1	0	0	1	0
Mold leachate, pH = 4.5, day 10	0	0	1	0
Mold leachate, pH = 4.5, day 40	1	1	1	1
Freeze/thaw, 5 cycles	0	0	1	1
Freeze/thaw, 15 cycles	0	0	1	1
Uniaxial strength	1	1	6	4

*FBHE, Fluidized Bed Heat Exchanger
+BH, Baghouse
=1, quantity of samples for testing

APPENDIX B

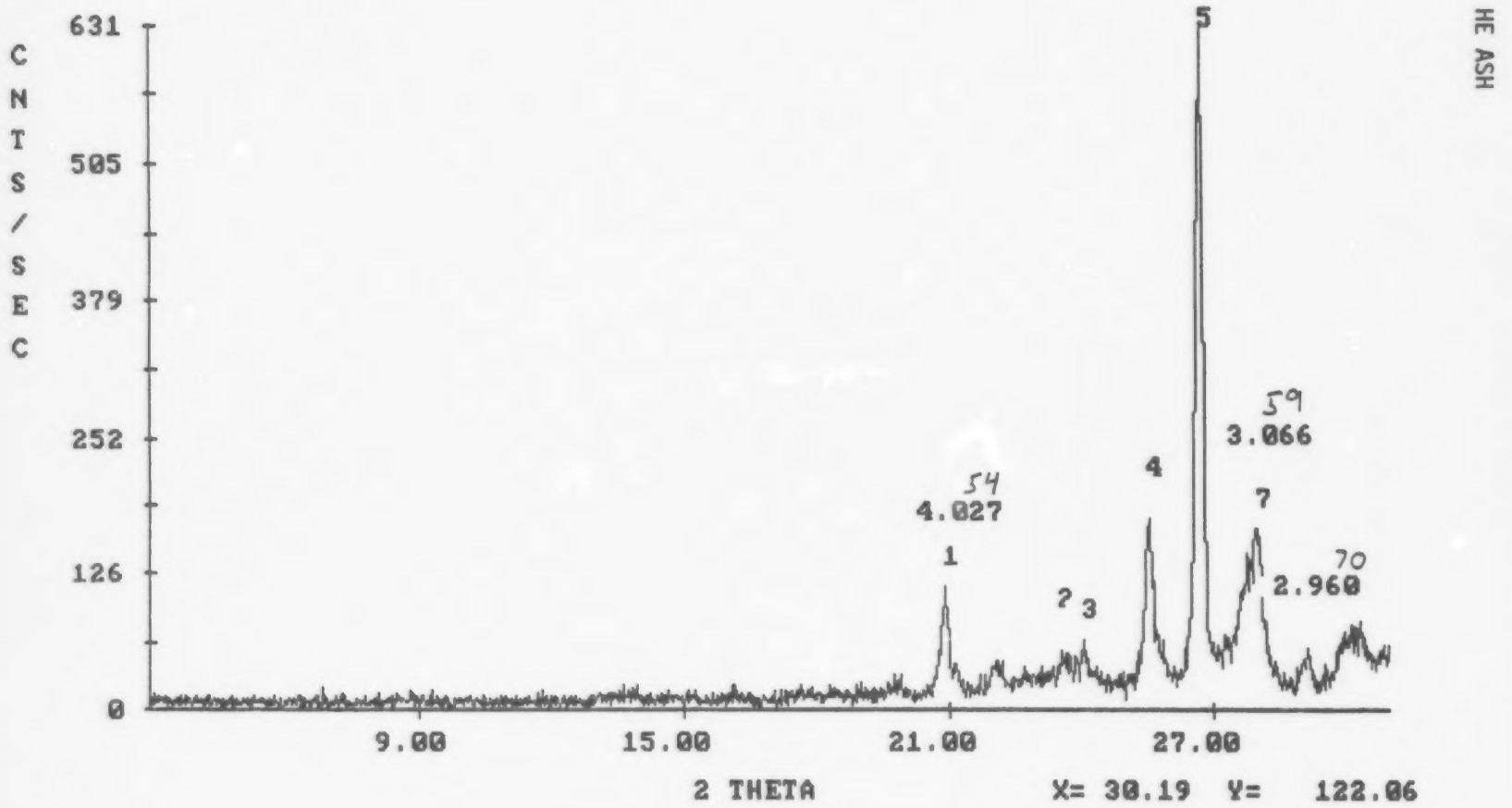
Raw Data From XRD Tests

3903 6-16-89 RPC1703.
START ANGLE= 3.000 DELTA 2THETA= .020 TIME= 1.0 TUBE=CU LAMDA= 1.542
SM= 0 BKGR= 0 0 0 KA2=0 TUBE=CU SCH= 9 SENS=5.00 CENTROID= 7 NTP= 5

N	2THETA	DSPACE	(1/D**2)	HEIGHT	AREA	P	W2	W4
1	20.916	4.2469	.0554	115.	13.	58.	.141	.116
2	23.541	3.7791	.0700	56.	7.	15.	.164	.133
3	24.058	3.6990	.0731	65.	8.	28.	.141	.118
4	25.536	3.4881	.0822	180.	32.	148.	.211	.176
5	26.693	3.3395	.0897	631.	96.	591.	.188	.152
6	27.762	3.2133	.0968	147.	18.	24.	.141	.125
7	27.997	3.1869	.0985	168.	33.	100.	.211	.165
8	31.361	2.8523	.1229	187.	42.	150.	.258	.224
9	35.618	2.5205	.1574	146.	23.	48.	.188	.160
10	36.581	2.4564	.1657	86.	17.	48.	.235	.192
11	38.641	2.3300	.1842	54.	7.	16.	.164	.124
12	39.503	2.2811	.1922	67.	7.	20.	.117	.098
13	40.903	2.2063	.2054	53.	6.	18.	.141	.117
14	42.442	2.1297	.2205	63.	9.	22.	.164	.135
15	43.003	2.1033	.2261	73.	9.	18.	.141	.127
16	45.076	2.0112	.2472	34.	5.	18.	.188	.157
17	49.221	1.8511	.2918	49.	6.	18.	.141	.116
18	50.178	1.8180	.3026	89.	13.	43.	.164	.149

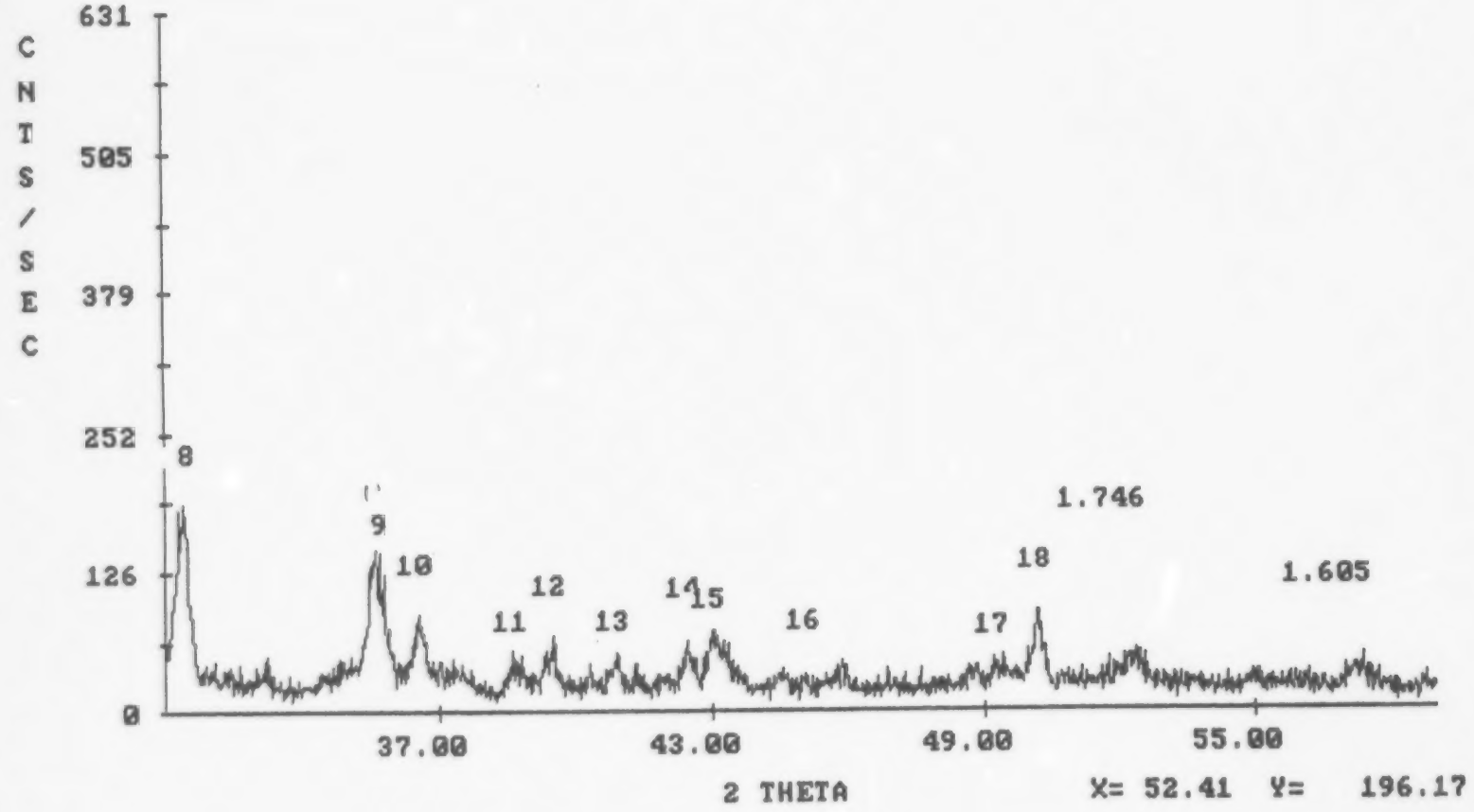
HE ASH

RPC170 6/16/89 S= .02 T= 1.0 RPC1703.



RPC170 6/16/89 S= .02 T= 1.0 RPC1703.

HE ASH



82

3903 6-18-89 RPC27074
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SM= 0 BKGR= 0 0 0 KA2=0 TUBE=CU SCH= 9 SENS=5.00 CENTROID= 7 NTP= 5

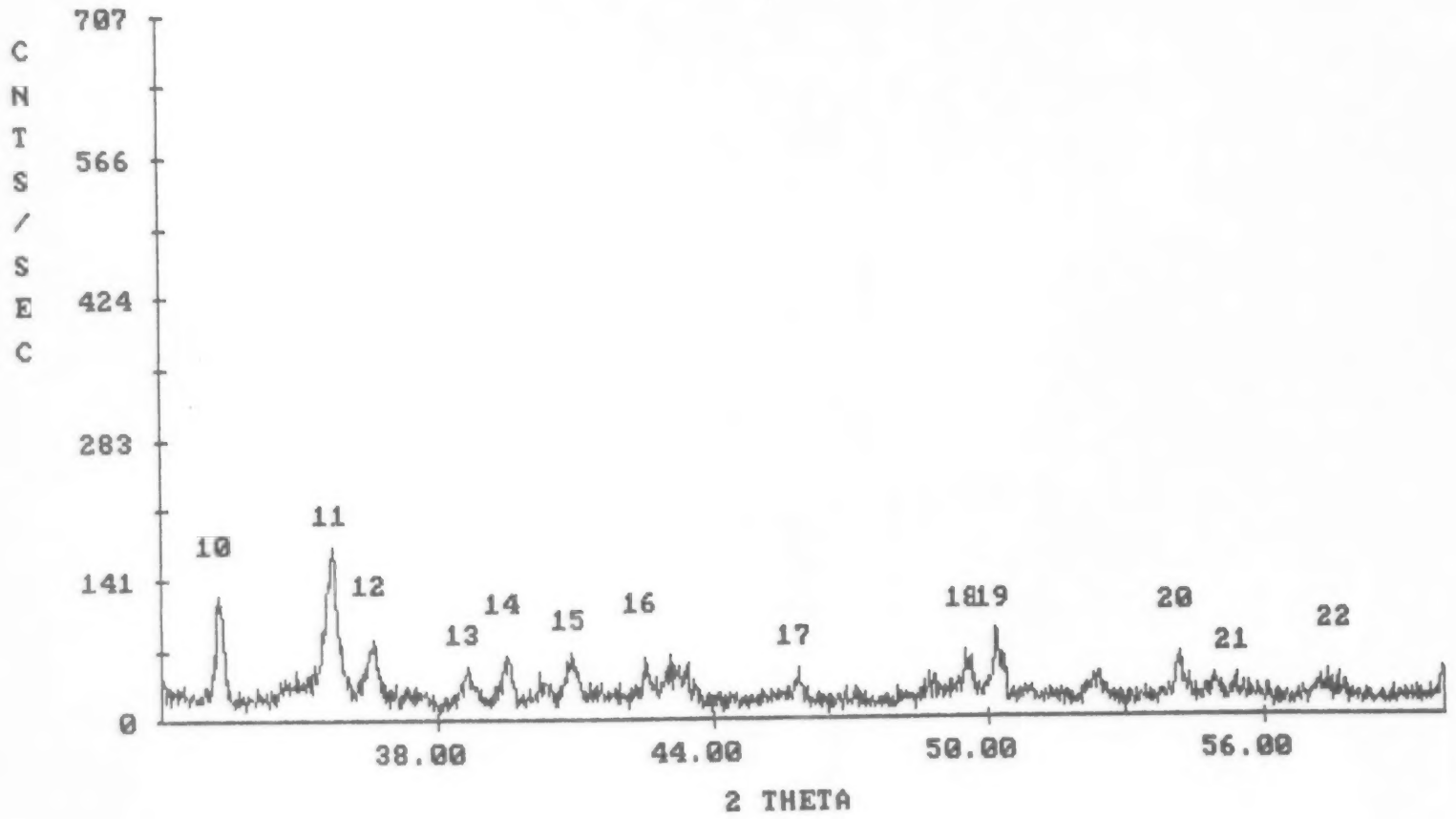
N	2THETA	DSPACE	(1/D**2)	HEIGHT	AREA	P	W2	W4
1	20.920	4.2461	.0555	132.	15.	78.	.141	.115
2	23.524	3.7817	.0699	55.	6.	15.	.117	.110
3	24.183	3.6802	.0738	68.	12.	29.	.211	.179
4	25.537	3.4880	.0822	228.	31.	154.	.164	.138
5	26.697	3.3390	.0897	707.	94.	630.	.164	.133
6	27.218	3.2762	.0932	71.	5.	13.	.094	.077
7	27.581	3.2339	.0956	87.	8.	13.	.094	.089
8	27.960	3.1909	.0982	146.	17.	42.	.141	.118
9	31.361	2.8523	.1229	117.	24.	82.	.235	.202
10	33.207	2.6978	.1374	124.	22.	116.	.211	.176
11	35.717	2.5137	.1583	175.	22.	68.	.141	.124
12	36.581	2.4563	.1657	82.	12.	37.	.164	.146
13	38.658	2.3290	.1844	53.	7.	18.	.164	.132
14	39.481	2.2824	.1920	63.	8.	20.	.141	.121
15	40.919	2.2054	.2056	68.	12.	28.	.211	.172
16	42.502	2.1269	.2211	62.	6.	23.	.117	.092
17	45.820	1.9803	.2550	51.	6.	24.	.164	.126
18	49.500	1.8413	.2949	67.	6.	15.	.117	.093
19	50.146	1.8191	.3022	88.	11.	37.	.141	.125
20	54.176	1.6929	.3489	63.	11.	44.	.211	.167
21	55.399	1.6584	.3636	43.	4.	14.	.141	.104
22	57.657	1.5987	.3913	43.	4.	12.	.117	.086

BH ASH

4101

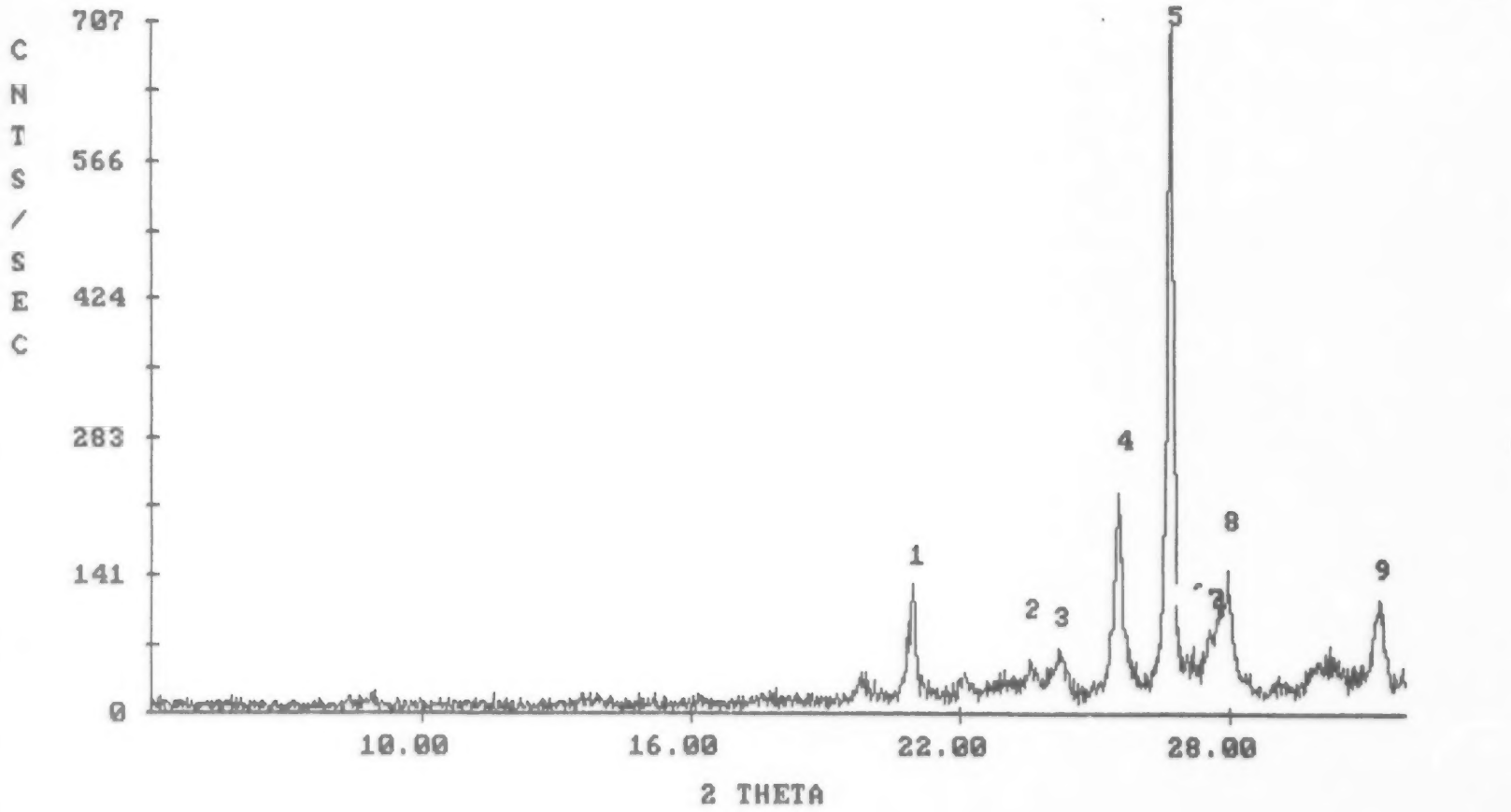
RPC270 6/18/89 S= .02 T= 1.0 RPC27074

BH ASH



RPC270 6/18/89 S= .02 T= 1.0 RPC27074

BH ASH



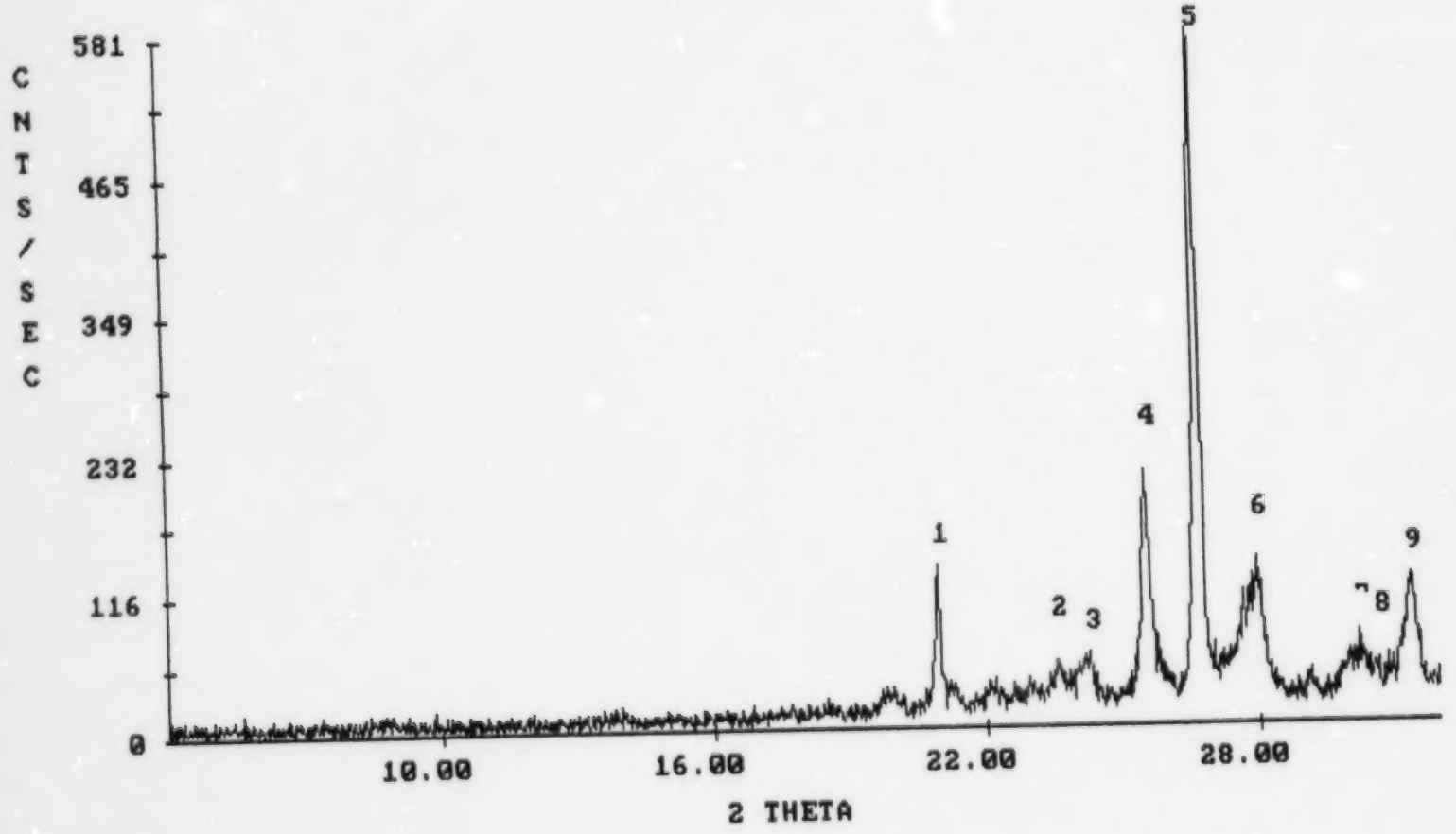
3903 6-17-89 RPC37075
START ANGLE= 3.000 DELTA 2THETA= .020 TIME= 1.0 TUBE=CU LAMDA= 1.542
SM= 0 BKGR= 0 0 0 KA2=0 TUBE=CU SCH= 9 SENS=5.00 CENTROID= 7 NTP= 5

N	2THETA	DSPACE	(1/D**2)	HEIGHT	AREA	P	W2	W4
1	20.917	4.2467	.0554	137.	15.	90.	.141	.110
2	23.505	3.7848	.0698	56.	10.	25.	.211	.185
3	24.276	3.6662	.0744	62.	7.	16.	.141	.118
4	25.504	3.4924	.0820	214.	30.	133.	.164	.141
5	26.683	3.3408	.0896	581.	90.	578.	.188	.154
6	27.943	3.1929	.0981	138.	29.	78.	.258	.211
7	30.221	2.9572	.1143	78.	6.	11.	.094	.079
8	30.655	2.9164	.1176	53.	7.	12.	.141	.126
9	31.343	2.8538	.1228	123.	21.	59.	.188	.169
10	33.256	2.6939	.1378	132.	12.	58.	.117	.093
11	35.643	2.5188	.1576	194.	21.	60.	.141	.107
12	35.678	2.5164	.1579	141.	12.	13.	.094	.083
13	36.618	2.4539	.1661	82.	10.	19.	.141	.116
14	38.625	2.3310	.1840	54.	6.	13.	.141	.103
15	39.541	2.2790	.1925	63.	12.	48.	.211	.185
16	40.958	2.2034	.2060	57.	7.	15.	.141	.115
17	42.477	2.1281	.2208	60.	7.	26.	.141	.119
18	42.962	2.1052	.2256	51.	6.	11.	.141	.127
19	43.418	2.0841	.2302	58.	5.	15.	.117	.088
20	48.721	1.8689	.2863	44.	6.	12.	.164	.130
21	49.503	1.8412	.2950	65.	7.	23.	.141	.107
22	50.198	1.8173	.3028	95.	14.	53.	.164	.148
23	51.441	1.7763	.3169	105.	5.	33.	.117	.043
24	54.141	1.6939	.3485	47.	6.	15.	.141	.126
25	54.940	1.6712	.3581	48.	5.	15.	.141	.108
26	55.979	1.6426	.3706	35.	4.	14.	.141	.113

MIX 1 ASH

RPC370 6/17/89 S= .02 T= 1.0 RPC37075

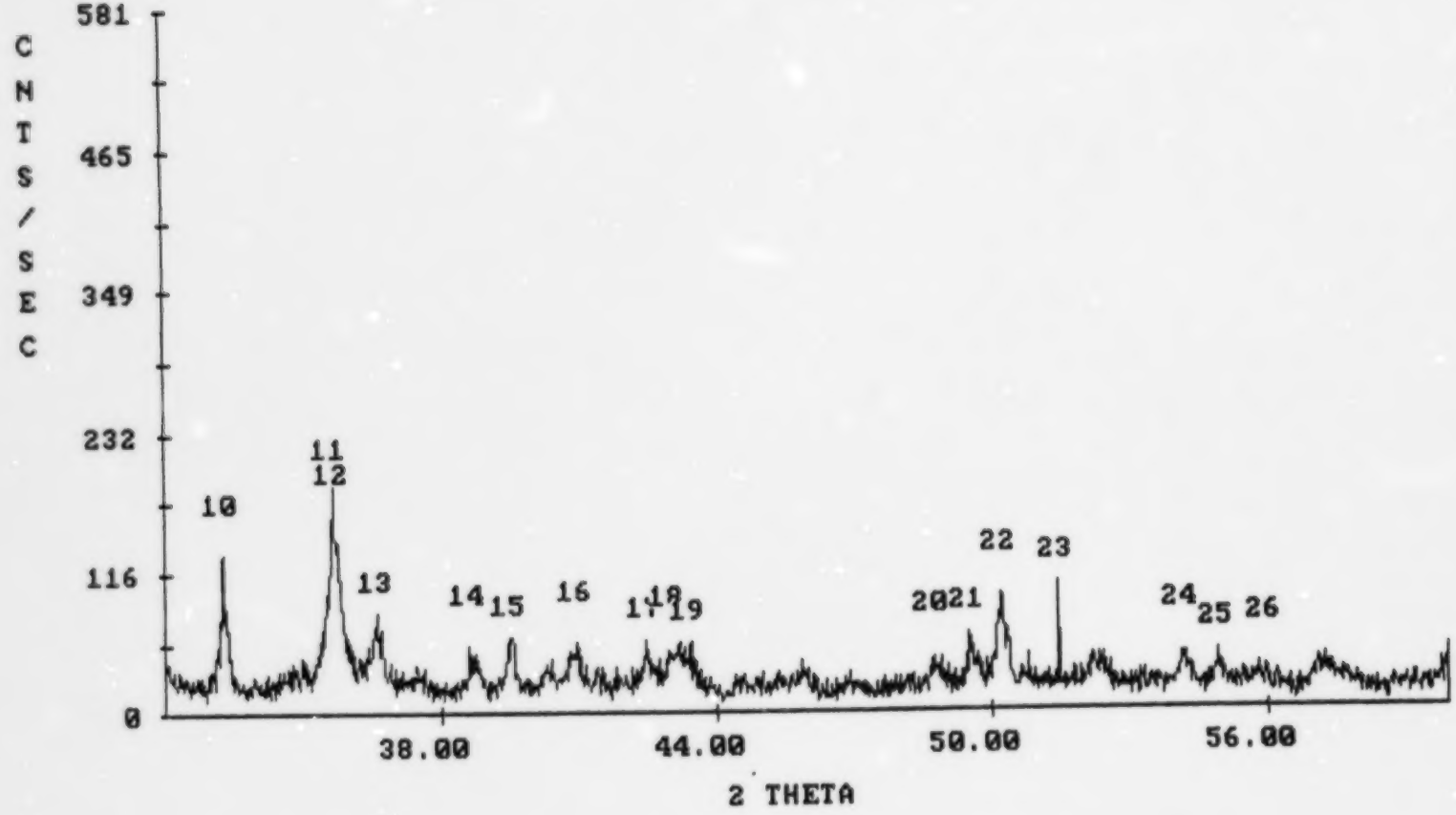
MIX 1 ASH



87

RPC370 6/17/89 S= .02 T= 1.0 RPC37075

MIX 1 ASH



3903 6-17-89 RPC47076
START ANGLE= 3.000 DELTA 2THETA= .020 TIME= 1.0 TUBE=CU LAMDA= 1.542
SM= 0 BKGR= 0 0 0 KA2=0 TUBE=CU SCH= 9 SENS=5.00 CENTROID= 7 NTP= 5

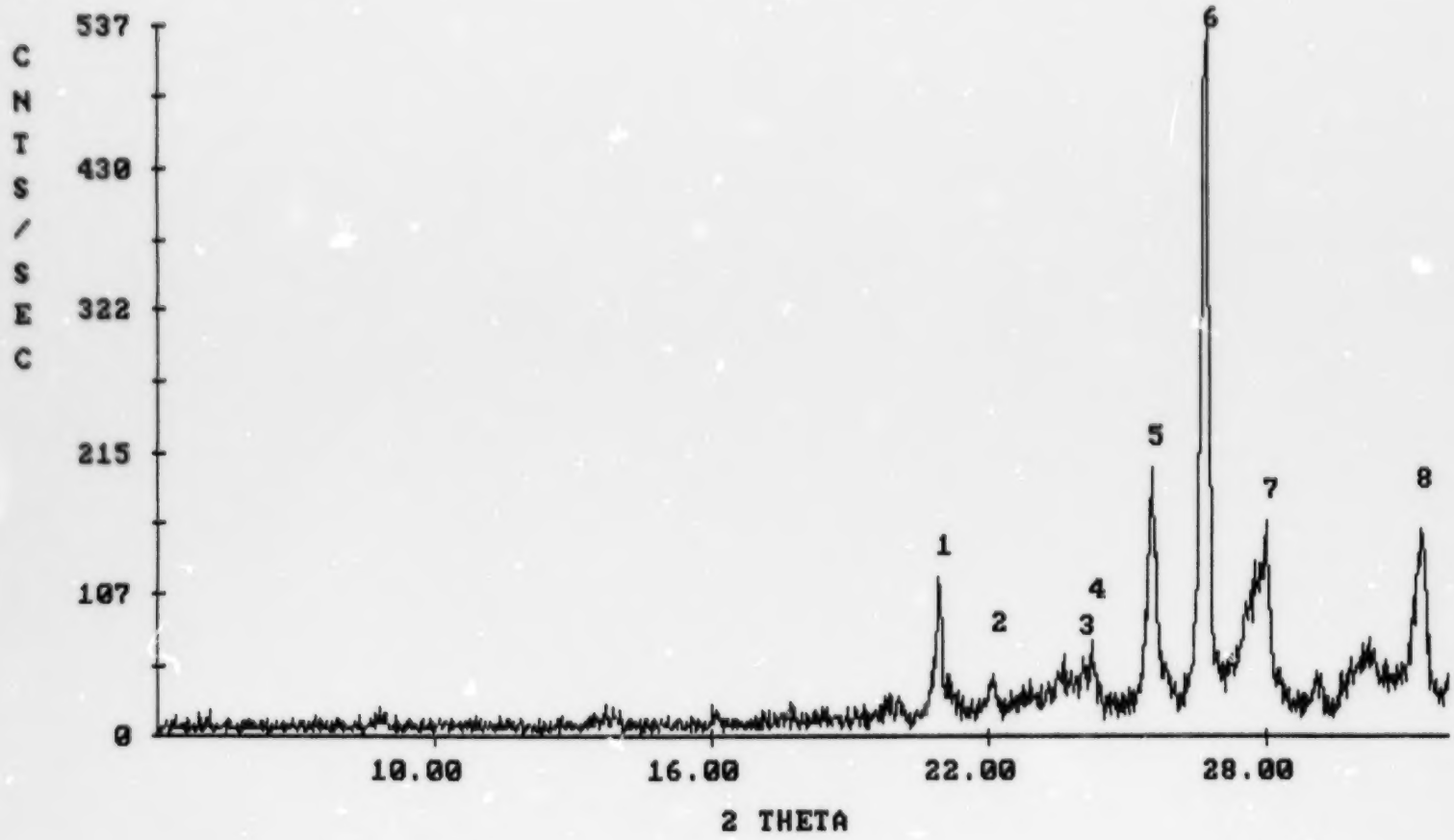
N	2THETA	DSPACE	(1/D**2)	HEIGHT	AREA	P	W2	W4
1	20.902	4.2498	.0554	121.	14.	78.	.141	.113
2	22.094	4.0231	.0618	46.	7.	23.	.188	.161
3	24.006	3.7068	.0728	59.	6.	13.	.117	.102
4	24.200	3.6775	.0739	71.	8.	23.	.141	.109
5	25.503	3.4926	.0820	204.	31.	139.	.188	.150
6	26.693	3.3395	.0897	537.	84.	496.	.188	.157
7	27.999	3.1867	.0985	164.	22.	74.	.164	.137
8	31.362	2.8522	.1229	157.	33.	135.	.235	.208
9	33.239	2.6953	.1377	76.	15.	64.	.235	.195
10	35.680	2.5163	.1579	164.	30.	101.	.211	.183
11	36.581	2.4563	.1657	104.	11.	40.	.141	.104
12	38.758	2.3232	.1853	45.	5.	13.	.141	.121
13	39.502	2.2812	.1922	63.	10.	42.	.188	.163
14	40.935	2.2046	.2058	59.	6.	16.	.117	.106
15	42.501	2.1269	.2211	61.	6.	13.	.117	.098
16	43.019	2.1025	.2262	73.	10.	31.	.164	.140
17	45.766	1.9825	.2544	41.	6.	17.	.164	.142
18	48.718	1.8690	.2863	49.	6.	17.	.164	.124
19	50.198	1.8174	.3028	94.	13.	41.	.164	.133
20	59.138	1.5622	.4098	28.	3.	12.	.141	.117

MIX 2 ASH

9076

RPC470 6/17/89 S= .02 T= 1.0 RPC47076

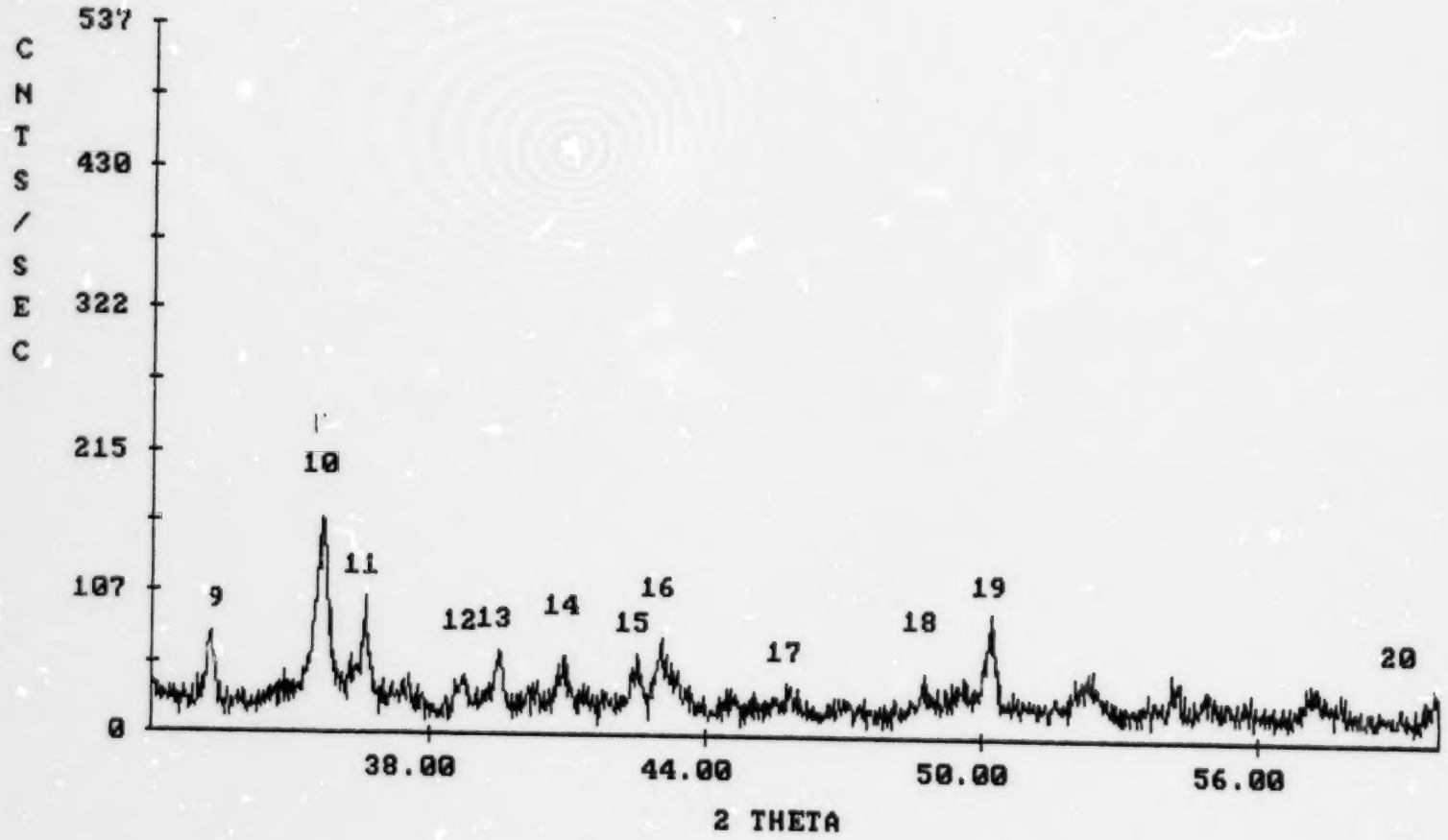
MIX 2 ASH



90

RPC470 6/17/89 S= .02 T= 1.0 RPC47076

MIX 2 ASH



APPENDIX C

Equilibrium Leach Test (ELT) Procedure

9.0 EQUILIBRIUM EXTRACTION

9.1 Apparatus

Drying oven set at 60 ± 3°C (in a fume hood).

Grinder capable of reducing sample to -100 mesh*.

ASTM No. 100 (150 um) sieve.

Balance, having a precision of ± 0.01 g.

250 mL plastic bottle
and/or
500 mL glass bottle with leak-proof screw closure.

Distilled water.

Thermometer, having a minimum temperature range from 10 to 30°C with a precision of ± 0.5°C.

MBS-design rotary extractor.

Vacuum or pressure filter with 0.45 um filter pads.

pH meter (with electrode).

30 mL pipette.

Desiccator.

9.2 Procedure

This test shall be started between 56 and 84 days after sample preparation. Crushed sample of the required mass from any type of mold may be used. For samples containing both organic and inorganic contaminants, Sections 9.2.1 and 9.2.2 shall both be carried out. It will be necessary to complete Method 4.0: Water Content before carrying out Section 9.2.2. If more than 7 days have elapsed since the water content determination, it should be repeated.

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* For organics, do not use a grinder which produces heat during the grinding operation, as this will cause sample drying. A mortar and pestle is good for this purpose.

9.2.1 Extraction of a Sample for Metals Analysis

Dry the sample to constant weight at 60 ± 5°C. Grind the dried sample to pass an ASTM No. 100 sieve. Label the 250 mL plastic bottle with the sample identification number and test number and rinse with distilled water. Weigh out approximately 40 g of the ground sample, recording the exact mass on Record Sheet 9, and place it in the bottle. Add a mass of distilled water at room temperature equal to four times the mass of solid for addition to the sample in the bottle. Record the room temperature and mass of the water on Record Sheet 9. Place the bottle in the rotary extractor and allow to tumble for 7 days ± 1 hour.

9.2.2 Extraction of a Sample for Organics Analysis

Grind the sample to approximately -100 mesh, using a mortar and pestle to minimize loss of volatile organics. Label the 500 mL glass bottle with the sample identification number and the test number. Place the wet equivalent of 80 g of dry sample* in the bottle, recording the exact mass on Record Sheet 9, and add a mass of distilled water at room temperature equal to four times the mass of dry solid. Record the room temperature and the mass of the water on Record Sheet 9. Place the bottle in the rotary extractor and allow to tumble for 7 days ± 1 hour.

9.2.3 Separation

For each sample bottle, assemble the filter holder and 0.45 um filter pad according to the manufacturer's instructions. Wet the filter pad with a small portion of the liquid from the bottle,

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* Wet weight = Dry weight/ (1 - Water content)

apply vacuum and transfer the extraction liquid to the filtration funnel. Take care to avoid transferring much of the solid from the bottle, because it may clog the filter pad. If the filter pad does become clogged, decant the extraction liquid remaining in the funnel back into the bottle, change the filter pad, and repeat the above process until enough of the extraction liquid has been collected for analysis. The extraction liquid may be collected in any container suitable for use with the filtration apparatus. The solid residue may be discarded.

Rinse the bottle originally containing the sample and extraction solution, and decant the extraction liquid from the collection vessel back into the cleaned bottle. The bottle must be labelled with the sample identification number and the test number. Measure the pH of the solution in each sample bottle and record the values on Record Sheet 9. The plastic bottle is for metals analysis, and the glass bottle is for organics analysis.

Pressure filtration may also be used.

9.3 Calculations

Concentration of contaminant in extraction liquid (moles/L)
= Concentration in extraction liquid (mg/L)
x 1 g/1000 mg
x 1/Molecular weight of contaminant (g/mole)

Amount of contaminant leached per gram of waste (mg/kg dry waste)
= Concentration in extraction liquid (mg/L)
x Mass of water (g)
x 1/Mass of sample (g)

APPENDIX D

ASTM Standard D 698-78 Method A "Test Methods for Moisture-Density Relations of Soils and Soil-Aggregate Mixtures, Using 5.5-lb (2.49-kg) Rammer and 12-in. (304.8-mm) Drop"

Designation: D 698 – 78

An American National Standard

Standard Test Methods for MOISTURE-DENSITY RELATIONS OF SOILS AND SOIL-AGGREGATE MIXTURES USING 5.5-lb (2.49-kg) RAMMER AND 12-in. (305-mm) DROP[1]

This standard is issued under the fixed designation D 698; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

These methods have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards

1. Scope

1.1 These laboratory compaction methods cover the determination of the relationship between the moisture content and density of soils and soil-aggregate mixtures (Note 1) when compacted in a mold of a given size with a 5.5-lb (2.49-kg) rammer dropped from a height of 12 in. (305 mm) (Note 2). Four alternative procedures are provided as follows:

1.1.1 *Method A*—A 4-in. (101.6-mm) mold; material passing a No. 4 (4.75-mm) sieve;

1.1.2 *Method B*—A 6-in. (152.4-mm) mold; material passing a No. 4 (4.75-mm) sieve;

1.1.3 *Method C*—A 6-in. (152.4-mm) mold; material passing a ¾-in. (19.0-mm) sieve; and

1.1.4 *Method D*—A 6-in. (152.4-mm) mold; material passing a ¾-in. (19.0-mm) sieve, corrected by replacement for material retained on a ¾-in. sieve.

NOTE 1—Soils and soil-aggregate mixtures should be regarded as natural occurring fine- or coarse-grained soils or composites or mixtures of natural soils, or mixtures of natural and processed soils or aggregates such as silt, gravel, or crushed rock.

NOTE 2—These laboratory compaction test methods when used on soils and soil-aggregates which are not free-draining will, in most cases, establish a well-defined optimum moisture content and maximum density (see Section 7). However, for free-draining soils and soil-aggregate mixtures, these methods will not, in many cases, produce a well-defined moisture-density relationship and the maximum density obtained will generally be less than that obtained by vibratory methods.

1.2 The method to be used should be indicated in the specifications for the material being tested. If no method is specified, the provisions of Section 5 shall govern.

2. Applicable Documents

2.1 *ASTM Standards:*

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]

D 854 Test Method for Specific Gravity of Soils[3]

D 2168 Methods for Calibration of Laboratory Mechanical-Rammer Soil Compactors[3]

D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures[3]

D 2487 Test Method for Classification of Soils for Engineering Purposes[3]

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[3]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

3. Apparatus

3.1 *Molds*—The molds shall be cylindrical in shape, made of rigid metal and be within the capacity and dimensions indicated in 3.1.1 or 3.1.2. The molds may be the "split" type, consisting either of two half-round sections, or a

[1] These methods are under the jurisdiction of ASTM Committee D-18 on Soil and Rock.

Current edition approved April 27, 1978. Published July 1978. Originally published as D 698 – 42 T. Last previous edition D 698 – 70.

[2] *Annual Book of ASTM Standards*, Vols 04.02 and 04.03.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vols 04.01, 04.02, 04.06, 05.05, and 14.02.

section of pipe split along one element, which can be securely locked together to form a cylinder meeting the requirements of this section. The molds may also be the "taper" type, providing the internal diameter taper is uniform and is not more than 0.200 in./linear ft (16.7 mm/linear m) of mold height. Each mold shall have a base plate assembly and an extension collar assembly, both made of rigid metal and constructed so they can be securely attached to or detached from the mold. The extension collar assembly shall have a height extending above the top of the mold of at least 2 in. (50.8 mm), which may include an upper section that flares out to form a funnel provided there is at least a 1/4-in. (19-mm) straight cylindrical section beneath it.

3.1.1 *Mold*, 4.0 in. (101.6 mm) in diameter, having a capacity of 1/30 ± 0.0004 ft^3 (944 ± 11 cm^3) and conforming to Fig. 1.

3.1.2 *Mold*, 6.0 in. (152.4 mm) in diameter, having a capacity of 1/13.333 ± 0.0009 ft^3 (2124 ± 25 cm^3) and conforming to Fig. 2.

3.1.3 The average internal diameter, height, and volume of each mold shall be determined before initial use and at intervals not exceeding 1000 times the mold is filled. The mold volume shall be calculated from the average of at least six internal diameter and three height measurements made to the nearest 0.001 in. (0.02 mm), or from the amount of water required to completely fill the mold, corrected for temperature variance in accordance with Table 1. If the average internal diameter and volume are not within the tolerances shown in Figs. 1 or 2, the mold shall not be used. The determined volume shall be used in computing the required densities.

3.2 *Rammer*—The rammer may be either manually operated (see 3.2.1) or mechanically operated (see 3.2.2). The rammer shall fall freely through a distance of 12.0 ± 1/16 in. (304.8 ± 1.6 mm) from the surface of the specimen. The manufactured weight of the rammer shall be 5.5 ± 0.02 lb (2.49 ± 0.01 kg). The specimen contact face shall be flat.

3.2.1 *Manual Rammer*—The specimen contact face shall be circular with a diameter of 2.000 ± 0.005 in. (50.80 ± 0.13 mm). The rammer shall be equipped with a guidesleeve which shall provide sufficient clearance so that the free fall of the rammer shaft and head will not be restricted. The guidesleeve shall have four vent holes at each end (eight holes total) located with centers 3/4 ± 1/16 in. (19.0 ± 1.6 mm) from each end and spaced 90 deg apart. The minimum diameter of the vent holes shall be 3/8 in. (9.5 mm).

3.2.2 *Mechanical Rammer*—The rammer shall operate mechanically in such a manner as to provide uniform and complete coverage of the specimen surface. There shall be 0.10 ± 0.03 in. (2.5 ± 0.8 mm) clearance between the rammer and the inside surface of the mold at its smallest diameter. When used with the 4.0-in. (101.6-mm) mold, the specimen contact face shall be circular with a diameter of 2.000 ± 0.005 in. (50.80 ± 0.13 mm). When used with the 6.0-in. (152.4-mm) mold, the specimen contact face shall have the shape of a section of a circle of a radius equal to 2.90 ± 0.02 in. (73.7 ± 0.5 mm). The sector face rammer shall operate in such a manner that the vertex of the sector is positioned at the center of the specimen. The mechanical rammer shall be calibrated and adjusted, as necessary, in accordance with 3.2.3.

3.2.3 *Calibration and Adjustment*—The mechanical rammer shall be calibrated, and adjusted as necessary, before initial use; near the end of each period during which the mold was filled 1000 times; before reuse after anything, including repairs, which may affect the test results significantly; and whenever the test results are questionable. Each calibration and adjustment shall be in accordance with Methods D 2168.

3.3 *Sample Extruder* (optional)—A jack, frame or other device adapted for the purpose of extruding compacted specimens from the mold.

3.4 *Balances*—A balance or scale of at least 20-kg capacity sensitive to ± 1 g and a balance of at least 1000-g capacity sensitive to ±0.01 g.

3.5 *Drying Oven*, thermostatically controlled, preferably of the forced-draft type, capable of maintaining a temperature of 230 ± 9°F (110 ± 5°C) for determining the moisture content of the compacted specimen.

3.6 *Straightedge*—A stiff metal straightedge of any convenient length but not less than 10 in. (254 mm). The scraping edge shall have a straightness tolerance of ±0.005 in. (±0.13 mm) and shall be beveled if it is thicker than 1/8 in.

(3 mm).

3.7 *Sieves*, 3-in. (75-mm), ¾-in. (19.0-mm) and No. 4 (4.75-mm), conforming to the requirements of Specification E 11.

3.8 *Mixing Tools*—Miscellaneous tools such as mixing pan, spoon, trowel, spatula, etc., or a suitable mechanical device for thoroughly mixing the sample of soil with increments of water.

4. Procedure

4.1 *Specimen Preparation*—Select a representative portion of quantity adequate to provide, after sieving, an amount of material weighing as follows: Method A—25 lb (11 kg); Methods B, C, and D—50 lb (23 kg). Prepare specimens in accordance with either 4.1.1 through 4.1.3 or 4.1.4.

4.1.1 *Dry Preparation Procedure*—If the sample is too damp to be friable, reduce the moisture content by drying until the material is friable; see 4.1.2. Drying may be in air or by the use of a drying apparatus such that the temperature of the sample does not exceed 140°F (60°C). After drying (if required), thoroughly break up the aggregations in such a manner as to avoid reducing the natural size of the particles. Pass the material through the specified sieve as follows: Methods A and B—No. 4 (4.75-mm); Methods C and D—¾-in. (19.0-mm). Correct for oversize material in accordance with Section 5, if Method D is specified.

4.1.2 Whenever practicable, soils classified as ML, CL, OL, GC, SC, MH, CH, OH and PT by Test Method D 2487 shall be prepared in accordance with 4.1.4.

4.1.3 Prepare a series of at least four specimens by adding increasing amounts of water to each sample so that the moisture contents vary by approximately 1½ %. The moisture contents selected shall bracket the optimum moisture content, thus providing specimens which, when compacted, will increase in mass to the maximum density and then decrease in density (see 7.2 and 7.3). Thoroughly mix each specimen to ensure even distribution of moisture throughout and then place in a separate covered container and allow to stand prior to compaction in accordance with Table 2. For the purpose of selecting a standing time, it is not required to perform the actual classification procedures described in Test Method D 2487 (except in the case of referee testing), if previous data exist which provide a basis for classifying the sample.

4.1.4 *Moist Preparation Method*—The following alternate procedure is recommended for soils classified as ML, CL, OL, GC, SC, MH, CH, OH, and PT by Test Method D 2487. Without previously drying the sample, pass it through the ¾-in. (19.0-mm) and No. 4 (4.75-mm) sieves. Correct for oversize material in accordance with Section 5, if Method D is specified. Prepare a series of at least four specimens having moisture contents that vary by approximately 1½ %. The moisture contents selected shall bracket the optimum moisture content, thus providing specimens which, when compacted, will increase in mass to the maximum density and then decrease in density (see 7.2 and 7.3). To obtain the appropriate moisture content of each specimen, the addition of a predetermined amount of water (see 4.1.3) or the removal of a predetermined amount of moisture by drying may be necessary. Drying may be in air or by the use of a drying apparatus such that the temperature of the specimen does not exceed 140°F (60°C). The prepared specimens shall then be thoroughly mixed and stand, as specified in 4.1.3 and Table 2, prior to compaction.

NOTE 3—With practice, it is usually possible to visually judge the point of optimum moisture closely enough so that the prepared specimens will bracket the point of optimum moisture content.

4.2 *Specimen Compaction*—Select the proper compaction mold, in accordance with the method being used, and attach the mold extension collar. Compact each specimen in three layers of approximately equal height. Each layer shall receive 25 blows in the case of the 4-in. (101.6-mm) mold; each layer shall receive 56 blows in the case of the 6-in. (152.4-mm) mold. The total amount of material used shall be such that the third compacted layer is slightly above the top of the mold, but not exceeding ¼ in. (6 mm). During compaction the mold shall rest on a uniform rigid foundation, such as provided by a cylinder or cube of concrete weighing not less than 200 lb (91 kg).

4.2.1 In operating the manual rammer, care shall be taken to avoid rebound of the rammer from the top end of the guidesleeve.

The guidesleeve shall be held steady and within 5 deg of the vertical. Apply the blows at a uniform rate not exceeding approximately 1.4 s per blow and in such a manner as to provide complete coverage of the specimen surface.

4.2.2 Following compaction, remove the extension collar; carefully trim the compacted specimen even with the top of the mold by means of the straightedge and determine the mass of the specimen. Divide the mass of the compacted specimen and mold, minus the mass of the mold, by the volume of the mold (see 3.1.3). Record the result as the wet density, γ_m, in pounds per cubic foot (or kilograms per cubic metre) of the compacted specimen.

4.2.3 Remove the material from the mold. Determine moisture content in accordance with Method D 2216, using either the whole specimen or alternatively a representative specimen of the whole specimen. The whole specimen must be used when the permeability of the compacted specimen is high enough so that the moisture content is not distributed uniformly throughout. If the whole specimen is used, break it up to facilitate drying. Obtain the representative specimen by slicing the compacted specimen axially through the center and removing 100 to 500 g of material from one of the cut faces.

4.2.4 Repeat 4.2 through 4.2.3 for each specimen prepared.

5. Oversize Corrections

5.1 If 30 % or more of the sample is retained on a 3/4-in. (19.0-mm) sieve, then none of the methods described under these methods shall be used for the determination of either maximum density or optimum moisture content.

5.2 *Methods A and B*—The material retained on the No. 4 (4.75-mm) sieve is discarded and no oversize correction is made. However, it is recommended that if the amount of material retained is 7 % or greater, Method C be used instead.

5.3 *Method C*—The material retained on the 3/4-in. (19.0-mm) sieve is discarded and no oversize correction is made. However, if the amount of material retained is 10 % or greater, it is recommended that Method D be used instead.

5.4 *Method D:*

5.4.1 This method shall not be used unless the amount of material retained on the 3/4-in. (19.0-mm) sieve is 10 % or greater. When the amount of material retained on the 3/4-in. sieve is less than 10 %, use Method C.

5.4.2 Pass the material retained on the 3/4-in. (19.0-mm) sieve through a 3-in. or 75-mm sieve. Discard the material retained on the 3-in. sieve. The material passing the 3-in. sieve and retained on the 3/4-in. sieve shall be replaced with an equal amount of material passing a 3/4-in. sieve and retained on a No. 4 (4.75-mm) sieve. The material for replacement shall be taken from an unused portion of the sample.

6. Calculations

6.1 Calculate the moisture content and the dry density of each compacted specimen as follows:

$$w = [(A - B)/(B - C)] \times 100$$

and

$$\gamma_d = [\gamma_m/(w + 100)] \times 100$$

where:

w = moisture content in percent of the compacted specimens,
A = mass of container and moist specimen,
B = mass of container and oven-dried specimen,
C = mass of container,
γ_d = dry density, in pounds per cubic foot (or kilograms per cubic metre) of the compacted specimen, and
γ_m = wet density, in pounds per cubic foot (or kilograms per cubic metre) of the compacted specimen.

7. Moisture-Density Relationship

7.1 From the data obtained in 6.1, plot the dry density values as ordinates with corresponding moisture contents as abscissas. Draw a smooth curve connecting the plotted points. Also draw a curve termed the "curve of complete saturation" or "zero air voids curve" on this plot. This curve represents the relationship between dry density and corresponding moisture contents when the voids are completely filled with water. Values of dry density and corresponding moisture contents for plotting the curve of complete saturation can be com-

puted using the following equation:

$$w_{sat} = [(62.4/\gamma_d) - (1/G_s)] \times 100$$

where:

w_{sat} = moisture content in percent for complete saturation,
γ_d = dry density in pounds per cubic foot (or kilograms per cubic metre),
G_s = specific gravity of the material being tested (see Note 4), and
62.4 = density of water in pounds per cubic foot (or kilograms per cubic metre).

NOTE 4—The specific gravity of the material can either be assumed or based on the weighted average values of: (*a*) the specific gravity of the material passing the No. 4 (4.75-mm) sieve in accordance with Test Method D 854; and (*b*) the apparent specific gravity of the material retained on the No. 4 sieve in accordance with Test Method C 127.

7.2 *Optimum Moisture Content*, w_o—The moisture content corresponding to the peak of the curve drawn as directed in 7.1 shall be termined the "optimum moisture content."

7.3 *Maximum Density*, γ_{max}—The dry density in pounds per cubic foot (or kilograms per cubic metre) of the sample at "optimum moisture content" shall be termed "maximum density."

8. Report

8.1 The report shall include the following.

8.1.1 Method used (Method A, B, C, or D).

8.1.2 Optimum moisture content.

8.1.3 Maximum density.

8.1.4 Description of rammer (whether manual or mechanical).

8.1.5 Description of appearance of material used in test, based on Practice D 2488 (Test Method D 2487 may be used as an alternative).

8.1.6 Origin of material used in test.

8.1.7 Preparation procedure used (moist or dry).

9. Precision

9.1 Criteria for judging the acceptability of the maximum density and optimum moisture content test results are given in Table 3. The standard deviation, s, is calculated from the equation:

$$s^2 = \frac{1}{n-1}\sum_{1}^{n}(x - \bar{x})^2$$

where:

n = number of determinations,
x = individual value of each determination, and
$\bar{x}$ = numerical average of the determinations.

9.2 Criteria for assigning standard deviation values for single-operator precision are not available at the present time.

TABLE 1 Volume of Water per Gram based on Temperature[4]

Temperature, °C (°F)	Volume of Water, ml/g
12 (53.6)	1.00048
14 (57.2)	1.00073
16 (60.8)	1.00103
18 (64.4)	1.00138
20 (68.0)	1.00177
22 (71.6)	1.00221
24 (75.2)	1.00268
26 (78.8)	1.00320
28 (82.4)	1.00375
30 (86.0)	1.00435
32 (89.6)	1.00497

[4] Values other than shown may be obtained by referring to the *Handbook of Chemistry and Physics*, Chemical Rubber Publishing Co., Cleveland, Ohio.

TABLE 2 Dry Preparation Method—Standing Times

Classification D 2487	Minimum Standing Time, h
GW, GP, SW, SP	no requirement
GM, SM	3
ML, CL, OL, GC, SC	18
MH, CH, OH, PT	36

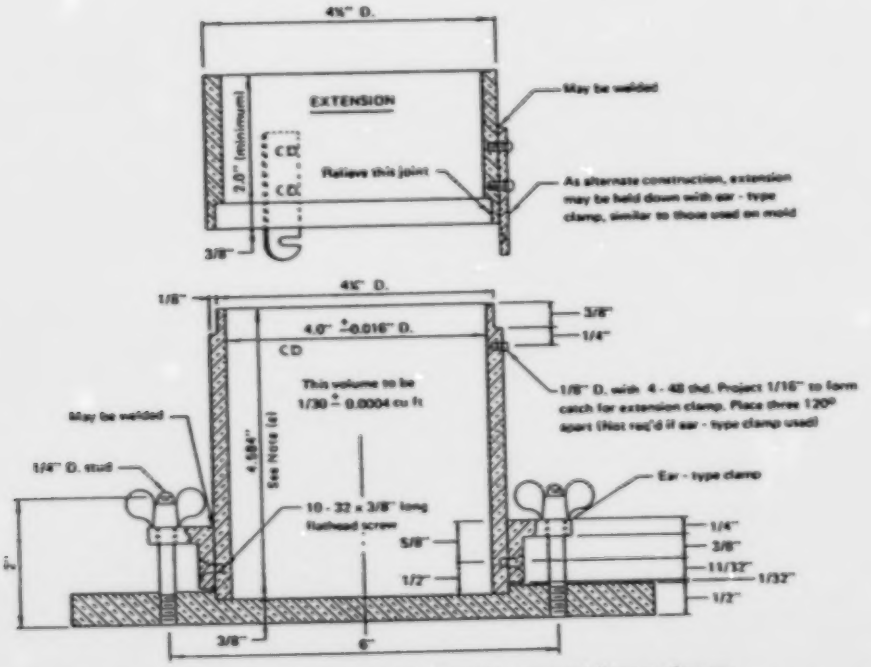
TABLE 3 Precision

	Standard Deviation, s	Acceptable Range of Two Results, Expressed as Percent of Mean Value[a]
Single-operator precision:		
Maximum density	...	1.9
Optimum moisture content	...	9.5
Multilaboratory precision:		
Maximum density	±1.66	4.0
Optimum moisture content	±0.86	15.0

[a] This column indicates a limiting range of values which should not be exceeded by the difference between any two results, expressed as a percentage of the average value. In cooperative test programs it has been determined that 95 % of the tests do not exceed the limiting acceptable ranges shown below. All values shown in this table are based on average test results from a variety of different soils and are subject to future revision.

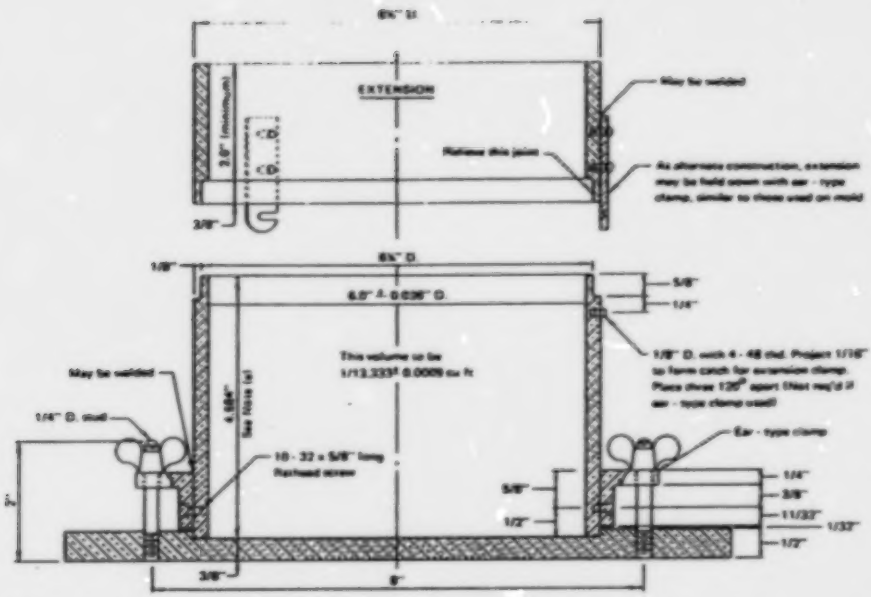
TABLE 4 Metric Equivalents for Figs. 1 and 2

in.	mm
0.016	0.41
0.026	0.66
1/32	0.80
1/16	1.6
1/8	3.2
1/4	6.4
11/32	8.7
3/8	9.5
1/2	12.7
5/8	15.9
2	50.8
2 1/2	63.5
4	101.6
4 1/4	108.0
4 1/2	114.3
4.584	116.43
6	152.4
6 1/2	165.1
8	203.2
ft³	**cm³**
1/30	944
0.004	11
1/13.333	2124
0.0009	25



NOTE 1—The tolerance on the height is governed by the allowable volume and diameter tolerances.
NOTE 2—The methods shown for attaching the extension collar to the mold and the mold to the base plate are recommended. However, other methods are acceptable, providing the attachments are equally as rigid as those shown.

FIG. 1 Cylindrical Mold, 4.0-in. for Soil Tests (see Table 4 for metric equivalents).



NOTE 1—The tolerance on the height is governed by the allowable volume and diameter tolerances.

NOTE 2—The methods shown for attaching the extension collar to the mold and the mold to the base plate are recommended. However, other methods are acceptable, providing the attachments are equally as rigid as those shown.

FIG. 2 Cylindrical Mold, 6.0-in. for Soil Tests (see Table 4 for metric equivalents).

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.

APPENDIX E

ASTM Standard D 560-82 "Methods for Freezing-and-Thawing Tests of Compacted Soil-Cement Mixtures"

Designation: D 560 – 82

Standard Methods for FREEZING-AND-THAWING TESTS OF COMPACTED SOIL-CEMENT MIXTURES[1]

This standard is issued under the fixed designation D 560; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

These methods have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These methods cover procedures for determining the soil-cement losses, moisture changes, and volume changes (swell and shrinkage) produced by repeated freezing and thawing of hardened soil-cement specimens. The specimens are compacted in a mold, before cement hydration, to maximum density at optimum moisture content using the compaction procedure described in Test Methods D 558.

1.2 Two methods, depending on soil gradation, are covered for preparation of material for molding specimens and for molding specimens as follows:

	Sections
Method A, using soil material passing a No. 4 (4.75-mm) sieve. This method shall be used when 100 % of the soil sample passes the No. 4 (4.75-mm) sieve	5
Method B, using soil material passing a ¾-in. (19.0-mm) sieve. This method shall be used when part of the soil sample is retained on the No. 4 (4.75-mm) sieve	6

2. Applicable Documents

2.1 *ASTM Standards:*

C 150 Specification for Portland Cement[2]

C 595 Specification for Blended Hydraulic Cements[3]

D 558 Test Methods for Moisture-Density Relations of Soil-Cement Mixtures[2]

D 559 Methods for Wetting-and-Drying Tests of Compacted Soil-Cement Mixtures[3]

D 2168 Methods for Calibration of Laboratory Mechanical-Soil Compactors[3]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

3. Significance and Use

3.1 These methods are used to determine the resistance of compacted soil-cement specimens to repeated freezing and thawing. These methods were developed to be used in conjunction with Methods D 559 and criteria given in the *Soil-Cement Laboratory Handbook*[5] to determine the minimum amount of cement required in soil-cement to achieve a degree of hardness adequate to resist field weathering.

4. Apparatus

4.1 *Mold*—A cylindrical metal mold having a capacity of 1/30 ± 0.0004 ft³ (944 ± 11 cm³) with an internal diameter of 4.0 ± 0.016 in. (101.60 ± 0.41 mm) and conforming to Fig. 1 to permit preparing compacted specimens of soil-cement mixtures of this size. The mold shall be provided with a detachable collar assembly approximately 2½ in. (63.5 mm) in height. The mold may be of the split type consisting of two half-round sections or a section of pipe with one side split perpendicular to the pipe circumference and that can be securely locked in place to form a closed cylinder

[1] These methods are under the jurisdiction of ASTM Committee D-18 on Soil and Rock and are the direct responsibility of Subcommittee D18.15 on Stabilization of Additives.

Current edition approved July 30, 1982. Published September 1982. Originally published as D 560 – 39. Last previous edition D 560 – 57 (1976).

[2] *Annual Book of ASTM Standards*, Vols 04.01 and 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vols 04.01, 04.02, 04.06, 05.05, and 14.02.

[5] *Soil-Cement Laboratory Handbook*, Portland Cement Assn., 1971.

having the dimensions described above. The mold and collar assembly shall be so constructed that it can be fastened firmly to a detachable base.

4.2 *Rammer:*

4.2.1 *Manual Rammer*—A manually operated metal rammer having a 2.0 ± 0.005-in. (50.80 ± 0.13-mm) diameter circular face and weighing 5.5 ± 0.02 lb (2.49 ± 0.01 kg). The rammer shall be equipped with a suitable guidesleeve to control the height of drop to a free fall of 12 ± 1/16 in. (304.8 ± 1.6 mm) above the elevation of the soil-cement. The guidesleeve shall have at least four vent holes not smaller than 3/8 in. (9.5 mm) spaced 90° apart and located with centers 3/4 ± 1/16 in. (19.0 ± 1.6 mm) from each end and shall provide sufficient clearance that free-falls of the rammer shaft and head will not be restricted.

4.2.2 *Mechanical Rammer*—A mechanically operated metal rammer having a 2.0 ± 0.005-in. (50.80 ± 0.13-mm) diameter face and a manufactured weight of 5.5 ± 0.02 lb (2.49 ± 0.01 kg). The operating weight of the rammer shall be determined from a calibration in accordance with Methods D 2168. The rammer shall be equipped with a suitable arrangement to control the height of drop to a free-fall of 12.0 ± 1/16 in. (304.8 ± 1.6 mm) above the elevation of the soil-cement.

4.2.3 *Rammer Face*—Strength and resistance to freezing and thawing of specimens compacted with the sector face rammer may differ from that of specimens compacted with the circular face rammer. Therefore, the sector face rammer shall not be used unless previous tests on like soil-cement mixtures show that similar resistance to freezing and thawing is obtained with the two types of rammers.

4.3 *Sample Extruder*—A jack, lever frame, or other device adapted for the purpose of extruding compacted specimens from the mold. Not required when a split-type mold is used.

4.4 *Balances*—A balance or scale of at least 25-lb (11.3-kg) capacity sensitive to 0.01 lb (0.005 kg) and a balance of at least 1000-g capacity sensitive to 0.1 g.

4.5 *Drying Oven*—A thermostatically controlled drying oven capable of maintaining temperatures of 230 ± 9°F (110 ± 5°C) for drying moisture samples.

4.6 *Freezing Cabinet*—A freezing cabinet capable of maintaining temperatures of −10°F (−23°C) or lower.

4.7 *Moist Room*—A moist room or suitable covered container capable of maintaining a temperature of 70 ± 3°F (21 ± 1.7°C) and a relative humidity of 100 % for 7-day storage of compacted specimens and for thawing frozen specimens.

4.8 *Wire Scratch Brush*—A wire scratch brush made of 2 by 1/16-in. (50.8 by 1.588-mm) flat No. 26 gage (0.46 mm) wire bristles assembled in 50 groups of 10 bristles each and mounted to form 5 longitudinal rows and 10 transverse rows of bristles on a 7½ by 2½-in. (190 by 63.5-mm) hardwood block.

4.9 *Straightedge*—A rigid steel straightedge 12 in. (305 mm) in length and having one beveled edge.

4.10 *Sieves*—3-in. (75-mm), 3/4-in. (19.0-mm), and No. 4 (4.75-mm) sieves conforming to the requirements of Specification E 11.

4.11 *Mixing Tools*—Miscellaneous tools such as mixing pan, and trowel, or a suitable mechanical device for thoroughly mixing the soil with cement and water.

4.12 *Butcher Knife*—A butcher knife approximately 10 in. (250 mm) in length for trimming the top of the specimens.

4.13 *Scarifier*—A six-pronged ice pick or similar apparatus to remove the smooth compaction plane at the top of the first and second layers of the specimen.

4.14 *Container*—A flat, round pan, for moisture absorption by soil-cement mixtures, about 12 in. (305 mm) in diameter and 2 in. (50 mm) deep.

4.15 *Measuring Device*—A measuring device suitable for accurately measuring the heights and diameters of test specimens to the nearest 0.01 in. (0.2 mm).

4.16 *Pans and Carriers*—Suitable pans for handling materials and carriers or trays for handling test specimens.

4.17 *Absorptive Pads*—¼-in. (6-mm) thick felt pads, blotters, or similar absorptive material for placing between specimens and specimen carriers.

4.18 *Graduate*—A graduated cylinder of 250-mL capacity for measuring water.

4.19 *Moisture Cans*—Suitable containers for moisture samples.

5. Method A—Using Soil Material Passing a No. 4 (4.75-mm) Sieve

5.1 *Preparation of Material for Molding Specimens:*

5.1.1 Prepare the soil sample in accordance with Method A of Test Methods D 558.

5.1.2 Select a sufficient quantity of the soil prepared as described in 5.1.1 to provide two (Note 1) compacted specimens and required moisture samples.

NOTE 1: (*Optional*)—Usually only one specimen (identified as No. 2) is required for routine testing. The other specimen (identified as No. 1) is made for research work and for testing unusual soils.

5.1.3 Add to the soil the required amount of cement conforming to Specification C 150 or Specification C 595. Mix the cement and soil thoroughly to a uniform color.

5.1.4 Add sufficient potable water to raise the soil-cement mixture to optimum moisture content at time of compaction and mix thoroughly.

5.1.5 When the soil used is a heavy textured clayey material, compact the mixture of soil, cement, and water in a container to a depth of about 2 in. (50 mm) using the rammer described in 4.2 or similar hand tamper, cover, and allow to stand for not less than 5 min but not more than 10 min to aid dispersion of the moisture and to permit more complete absorption by the soil-cement.

5.1.6 After the absorption period, thoroughly break up the mixture, without reducing the natural size of individual particles, until it will pass a No. 4 (4.75-mm) sieve, as judged by eye, and then remix.

5.2 *Molding Specimens:*

5.2.1 Form a specimen by immediately compacting the soil-cement mixture in the mold, with the collar attached, and later trimming the specimen in the same manner as directed for Method A of Test Methods D 558, and in addition scarify the tops of the first and second layers to remove smooth compaction planes before placing and compacting the succeeding layers. This scarification shall form grooves at right angles to each other, approximately ⅛ in. (3.2 mm) in width and ⅛ in. (3.2 mm) in depth and approximately ¼ in. (6.4 mm) apart.

5.2.2 During compaction, take from the batch a representative sample of the soil-cement mixture, weighing not less than 100 g, weigh immediately and dry in an oven at 230 ± 9°F (110 ± 5°C) for at least 12 h or to constant weight. Calculate the percentage of moisture as directed in Test Methods D 558 to check against design moisture content.

5.2.3 Weigh the compacted specimen and mold, remove the specimen from the mold, and calculate the oven-dry weight of each specimen in lb/ft³ (g/cm³) to check against design density.

5.2.4 Identify the specimen on a metal tag (or other suitable device) as No. 1 (Note 1) together with other needed identification marks and use to obtain data on moisture and volume changes during the test.

5.2.5 Form a second specimen as rapidly as possible and determine the percentage of moisture and oven-dry weight as described in 5.2.1 to 5.2.3. Identify this specimen as No. 2 together with other needed identification marks and use to obtain data on soil-cement losses during the test.

5.2.6 Determine the average diameter and height of the No. 1 specimen and calculate its volume.

5.2.7 Place the specimens on suitable carriers in the moist room and protect them from free water for a period of 7 days.

5.2.8 Weigh and measure the No. 1 specimen at the end of the 7-day storage period to provide data for calculating its moisture content and volume.

NOTE 2—It is important that all height and diameter measurements be accurate to within 0.01 in. (0.2 mm) and be taken at the same points on the specimen at all times.

5.3 *Procedure:*

5.3.1 At the end of the storage in the moist room, place water-saturated felt pads about ¼ in. (6 mm) thick, blotters, or similar absorptive material between the specimens and the carriers, and place the assembly in a freezing cabinet having a constant temperature not warmer than −10°F (−23°C) for 24 h and remove. Weigh and measure the No. 1 specimen (volume and moisture change specimen).

5.3.2 Place the assembly in the moist room or suitable covered container having a temperature of 70°F (21°C) and a relative humidity of 100 % for 23 h and remove. Free potable water shall be made available to the absorbent pads under the specimens to permit the specimens to absorb water by capillary action during the thawing period. Weigh and measure the No. 1 specimen.

5.3.3 Give specimen No. 2 (soil-cement loss specimen) two firm strokes on all areas with the wire scratch brush. The brush shall be held

with the long axis of the brush parallel to the longitudinal axis of the specimen or parallel to the ends as required to cover all areas of the specimen. Apply these strokes to the full height and width of the specimen with a firm stroke corresponding to approximately 3-lbf (13.3-N) force (Note 3). Eighteen to twenty vertical brush strokes are required to cover the sides of the specimen twice and four strokes are required on each end.

NOTE 3—This pressure is measured as follows: clamp a specimen in a vertical position on the edge of a platform scale and zero the scale. Apply vertical brushing strokes to the specimen and note the force necessary to register approximately 3 lbf (13.3 N).

5.3.4 After being brushed, the specimens shall be turned over end for end before they are replaced on the water-saturated pads.

5.3.5 The procedures described in 5.3.1 to 5.3.4 constitute one cycle (48 h) of freezing and thawing. Again place the specimens in the freezing cabinet and continue the procedure for 12 cycles.

NOTE 4—Weight determinations of specimen No. 2 before and after brushing are usually made at the end of each cycle when conducting research and making special investigations. Some specimens made of silty and clayey soils tend to scale on sides and ends particularly after about the sixth cycle of test. This scale shall be removed with a sharp-pointed instrument such as an ice pick, since the regular brushing may not be effective.

5.3.6 The No. 1 specimen may be discontinued prior to 12 cycles should be measurements become inaccurate due to soil-cement loss of the specimen.

NOTE 5—If it is not possible to run the cycles continuously because of Sundays, holidays, or for any other reason, the specimens shall be held in the freezing cabinet during the layover period if possible.

5.3.7 After 12 cycles of test, dry the specimens to constant weight at 230°F (110°C) and weigh to determine the oven-dry weight of the specimens.

5.3.8 The data collected will permit calculations of volume and moisture changes of specimen No. 1 and the soil-cement losses of specimen No. 2 after the prescribed 12 cycles of test.

6. Method B—Using Material Passing a ¾-in. (19.0-mm) Sieve

6.1 *Preparation of Material for Molding Specimens:*

6.1.1 Prepare the soil sample in accordance with Method B of Test Methods D 558.

6.1.2 Select and maintain separate representative samples of soil passing the No. 4 (4.75-mm) sieve and of saturated, surface-dry aggregate passing the ¾-in. (19.0-mm) sieve and retained on the No. 4 (4.75-mm) sieve so that the total sample will be enough to provide two (Note 1) compacted specimens and required moisture samples. The percentage, by oven-dry weight, of aggregate passing the ¾-in. (19.0-mm) sieve and retained on the No. 4 (4.75-mm) sieve shall be the same as the percentage passing the 3-in. (75-mm) sieve and retained on the No. 4 (4.75-mm) sieve in the original sample.

6.1.3 Add to the sample passing the No. 4 (4.75-mm) sieve, the amount of cement conforming to Specification C 150, or Specification C 595, required for the total sample described in 6.1.2. Mix the cement and soil thoroughly to a uniform color.

6.1.4 Add to the sample passing the No. 4 (4.75-mm) sieve, sufficient water to raise the total soil-cement mixture specified in 6.1.2 to optimum moisture content at time of compaction and facilitate moisture dispersion as described for Method A in 5.1.4 to 5.1.6.

6.1.5 After preparation of the mixture as described in 6.1.1 to 6.1.4, add the saturated, surface-dry aggregate to the mixture and mix thoroughly.

6.2 *Molding Specimens:*

6.2.1 Form a specimen by immediately compacting the soil-cement mixture in the mold (with the collar attached) and later trimming the specimen in accordance with Method B of Test Methods D 558, and in addition as the mixture for each layer is placed in the mold, spade along the inside of the mold with a butcher knife before compaction to obtain uniform distribution of the material retained on the No. 4 (4.75-mm) sieve and scarify the tops of the first and second layers as described for Method A of these methods.

6.2.2 During compaction take from the batch a representative sample of the soil-cement mixture weighing not less than 500 g, weigh immediately, and dry in an oven at 230 ± 9°F (110 ± 5°C) at least 12 h or to constant weight to determine the moisture content to check against design moisture content.

6.2.3 Form a second specimen as rapidly as

possible in the same manner.

6.2.4 Weigh each compacted specimen to check against design density, identify, measure the No. 1 specimen (Note 1), place in the moist room, and measure the No. 1 specimen again at the end of the 7-day storage period as described for Method A in 5.2.3 to 5.2.8 (Note 2).

6.3 *Procedure*—Proceed as directed in Method A (see 5.3).

7. Calculations

7.1 Calculate the volume and moisture changes and the soil-cement losses of the specimens as follows:

7.1.1 Calculate the difference between the volume of specimen No. 1 at the time of molding and subsequent volumes as a percentage of the original volume.

7.1.2 Calculate the moisture content of specimen No. 1 at the time of molding and subsequent moisture contents as a percentage of the original oven-dry weight of the specimen.

7.1.3 Correct the oven-dry weight of specimen No. 2 as obtained in 5.3.7 for water that has reacted with the cement and soil during the test and is retained in the specimen at 230°F (110°C) as follows:

$$\text{Corrected oven-dry weight} = (A/B) \times 100$$

where:

A = oven-dry weight after drying at 230°F (110°C), and

B = percentage of water retained in specimen plus 100.

The percentage of water retained in specimen No. 2 after drying at 230°F (110°C) for use in the above formula can be assumed to be equal to the water retained in specimen No. 1. When No. 1 specimens are not molded, the foregoing data are not available and the average values prescribed in Table I are used.

7.1.4 Calculate the soil-cement loss of specimen No. 2 as a percentage of the original oven-dry weight of the specimen as follows:

$$\text{Soil-cement loss, \%} = (A/B) \times 100$$

where:

A = original calculated oven-dry weight minus final corrected oven-dry weight, and

B = original calculated oven-dry weight.

8. Report

8.1 The report shall include the following:

8.1.1 The designed optimum moisture and maximum density of the molded specimens,

8.1.2 The moisture content and density obtained in molded specimens.

NOTE 6—Good laboratory practice permits the following tolerances between design factors and those obtained in the molded specimens:

Moisture content	±1 percentage point
Density	±3 lb/ft³ (0.048 g/cm³)

8.1.3 The designed cement content, in percent, of the molded specimens,

8.1.4 The maximum volume change, in percent, and maximum moisture content during test of specimen No. 1, and

8.1.5 The soil-cement loss, in percent, of specimen No. 2.

9. Precision and Accuracy

9.1 Precision and accuracy of this test procedure have not been established. Data are available on the variability of weight losses for duplicate specimens. These data will be analyzed and a statement will be included in the next revision.

TABLE I Average Values

AASHTO Soil Classification	Average Water Retained After Drying at 230°F (110°C), %
A-1, A-3	1.5
A-2	2.5
A-4, A-5	2.0
A-6, A-7	3.5

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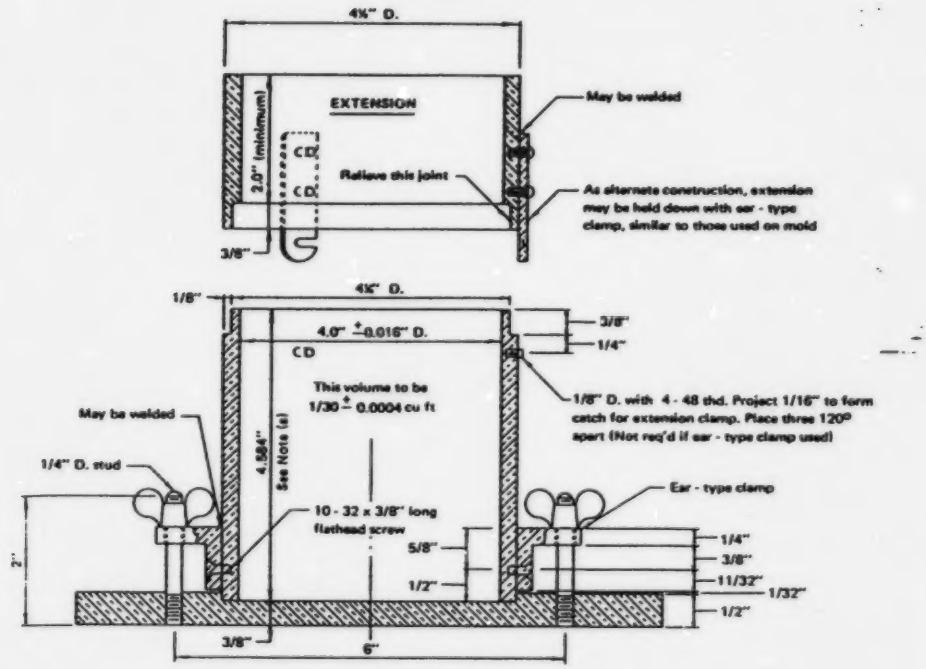

Metric Equivalents

in.	mm
0.016	0.41
0.026	0.66
1/32	0.80
1/16	1.6
1/8	3.2
1/4	6.4
11/32	8.7
3/8	9.5
1/2	12.7
5/8	15.9
2	50.8
2½	63.5
4	101.6
4¼	108.0
4½	114.3
4.584	116.43
6	152.4
6½	165.1
8	203.2
ft^3	**cm^3**
1/30	944
0.004	11
1/0.0047	2124
0.0009	25

NOTE (a)—The tolerance on the height as governed by the allowable volume and diameter tolerances.

NOTE (b)—The methods shown for attaching the extension collar to the mold and the mold to the base plate are recommended. However, other methods are acceptable, providing the attachments, are equally as rigid as those shown.

FIG. 1 Cylindrical Mold

APPENDIX F

ASTM Standard D 1633-84 "Compressive Strength of Molded Soil Cement Cylinders"

Designation: D 1633 – 84

Standard Test Method for COMPRESSIVE STRENGTH OF MOLDED SOIL-CEMENT CYLINDERS[1]

This standard is issued under the fixed designation D 1633; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method covers the determination of the compressive strength of soil-cement using molded cylinders as test specimens.

1.2 Two alternative procedures are provided as follows:

1.2.1 *Method A*—This procedure uses a test specimen 4.0 in. (101.6 mm) in diameter and 4.584 in. (116.4 mm) in height. Height to diameter ratio equals 1.15.

1.2.2 *Method B*—This procedure uses a test specimen 2.8 in. (71.1 mm) in diameter and 5.6 in. (142.2 mm) in height. Height to diameter ratio equals 2.00.

1.3 The values stated in inch-pound units are to be regarded as the standard.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 *ASTM Standards:*

C 42 Method of Obtaining and Testing Drilled Cores and Sawed Beams of Concrete[2]

D 559 Methods for Wetting-and-Drying Tests of Compacted Soil-Cement Mixtures[3]

D 560 Methods for Freezing-and-Thawing Tests of Compacted Soil-Cement Mixtures[3]

D 1632 Method of Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory[3]

E 4 Practices for Load Verification of Testing Machines[4]

3. Significance and Use

3.1 Method A makes use of the same compaction equipment and molds commonly available in soil laboratories and used for other soil-cement tests. It is considered that Method A gives a relative measure of strength rather than a rigorous determination of compressive strength. Because of the lesser height to diameter ratio (1.15) of the cylinders, the compressive strength determined by Method A will normally be greater than that for Method B.

3.2 Method B, because of the greater height to diameter ratio (2.00), gives a better measure of compressive strength from a technical viewpoint since it reduces complex stress conditions that may occur during the crushing of Method A specimens.

3.3 In practice, Method A has been more commonly used than Method B. As a result, it has been customary to evaluate or specify compressive strength values as determined by Method A. A factor for converting compressive

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[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.15 on Stabilization with Admixtures.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vols 03.01, 04.02, 07.01, and 08.03.

strength values based on height to diameter ratio is given in Section 7[3].

4. Apparatus

4.1 *Compression Testing Machine*—This machine may be of any type having sufficient capacity and control to provide the rate of loading prescribed in 6.2. It shall conform to the requirements of Section 15 of Practices E 4. The testing machine shall be equipped with two steel bearing blocks with hardened faces (Note 1), one of which is a spherically seated head block that normally will bear on the upper surface of the specimen, and the other a plain rigid block on which the specimen will rest. The bearing faces shall be at least as large, and preferably slightly larger, than the surface of the specimen to which the load is applied. The bearing faces, when new, shall not depart from a plane by more than 0.0005 in. (0.013 mm) at any point, and they shall be maintained within a permissible variation limit of 0.001 in. (0.02 mm). In the spherically seated block, the diameter of the sphere shall not greatly exceed the diameter of the specimen and the center of the sphere shall coincide with the center of the bearing face. The movable portion of this block shall be held closely in the spherical seat, but the design shall be such that the bearing face can be rotated freely and tilted through small angles in any direction.

NOTE 1—It is desirable that the bearing faces of blocks used for compression testing of soil-cement have a hardness of not less than 60 HRC.

4.2 *Molds and Compaction Equipment*, in accordance with Methods D 559 or Methods D 560 for Method A; Method D 1632 for Method B.

5. Test Specimens

5.1 Mold the test specimens as follows:

5.1.1 *Method A*—Specimens are 4.0 in. (101.6 mm) in diameter and 4.584 in. (116.4 mm) in height and are molded in accordance with Methods D 559 or Methods D 560.

5.1.2 *Method B*—Specimens are 2.8 in. (71.1 mm) in diameter and 5.6 in. (142.2 mm) in height and are molded in accordance with Method D 1632.

NOTE 2—These methods may be used for testing specimens of other sizes. If the soil sample includes material retained on the 4.75-mm (No. 4) sieve, it is recommended that Method A be used, or that larger test specimens, 4.0 in. (101.6 mm) in diameter and 8.0 in. (203.2 mm) in height, be molded in a manner similar to Method B.

5.2 Moist cure the specimens in accordance with Method D 1632.

5.3 At the end of the moist-cure period, immerse the specimens in water for 4 h.

5.4 Remove the specimens from the water and make compression tests as soon as practicable, keeping specimens moist by a wet burlap or blanket covering.

NOTE 3—Other conditioning procedures, such as air or oven drying, alternate wetting and drying, or alternate freezing and thawing may be specified after an initial moist curing period. Curing and conditioning procedures shall be given in detail in the report.

5.5 Check the smoothness of the faces with a straightedge. If necessary, cap the faces to meet the requirements of the section on Capping Specimens of Method D 1632.

6. Procedure

6.1 Place the lower bearing block on the table or platen of the testing machine directly under the spherically seated (upper) bearing block. Place the specimen on the lower bearing block, making certain that the vertical axis of the specimen is aligned with the center of thrust of the spherically seated block. As this block is brought to bear on the specimen, rotate its movable portion gently by hand so that uniform seating is obtained.

6.2 Apply the load continuously and without shock. A screw power testing machine, with the moving head operating at approximately 0.05 in. (1 mm)/min when the machine is running idle, may be used. With hydraulic machines, adjust the loading to a constant rate within the limits of 20 ± 10 psi (140 ± 70 kPa)/s, depending upon the strength of the specimen. Record the total load at failure of the test specimen to the nearest 10 lbf (40 N).

7. Calculation

7.1 Calculate the unit compressive strength of the specimen by dividing the maximum load by the cross-sectional area.

NOTE 4—If desired, make allowance for the ratio of height to diameter (h/d) by multiplying the compressive

[3] For additional discussion on the significance and use of compressive strength results, see the *Soil-Cement Laboratory Handbook*, Chapter 4, Portland Cement Association, Skokie, IL, 1971, pp 31 and 32.

strength of Method B specimens by the factor 1.10. This converts the strength for an h/d ratio of 2.00 to that for the h/d ratio of 1.15 commonly used in routine testing of soil-cement (see Section 3). This conversion is based on that given in Method C 42, which has been found applicable for soil-cement.

8. Report

8.1 The report shall include the following:

8.1.1 Specimen identification number,

8.1.2 Diameter and height, in. (mm),

8.1.3 Cross-sectional areas, in^2 (mm^2),

8.1.4 Maximum load, to the nearest 10 lbf (40 N),

8.1.5 Conversion factor for height to diameter ratio (see Note 4), if used,

8.1.6 Compressive strength, calculated to the nearest 5 psi (35 kPa),

8.1.7 Age of specimen, and

8.1.8 Details of curing and conditioning periods, and moisture content at the time of test.

9. Precision and Bias

9.1 The precision and bias of this test method have not been established by an interlaboratory test program. However, based on the test data that are available, the following may serve as a guide as to the variability of compressive strength test results.

9.1.1 Tests were performed in a single lab on 122 sets of duplicate specimens molded from 21 different soil materials. The average difference in strength on duplicate specimens was 8.1 % and the median difference was 6.2 %. These values are expressed as the percent of the average strength of the two specimens as follows:

$$\% \text{ Difference} = \frac{(\text{high value} - \text{low value})}{(\text{high value} + \text{low value})/2} \times 100$$

The distribution of the variation is shown in Fig. 1. The data[6,7] cover a wide range of cement contents and compressive strengths.

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[6] Packard, R. G., "Alternate Measures for Measuring Freeze-Thaw and Wet-Dry Resistance of Soil-Cement Mixtures," *Highway Research Bulletin*, 353, Transportation Research Board, 1962, pp 8–41.

[7] Packard, R. G., and Chapman, G. A., "Developments in Durability Testing of Soil-Cement Mixtures," Highway Research Record No. 36, Transportation Research Board, 1963, pp 97–122.

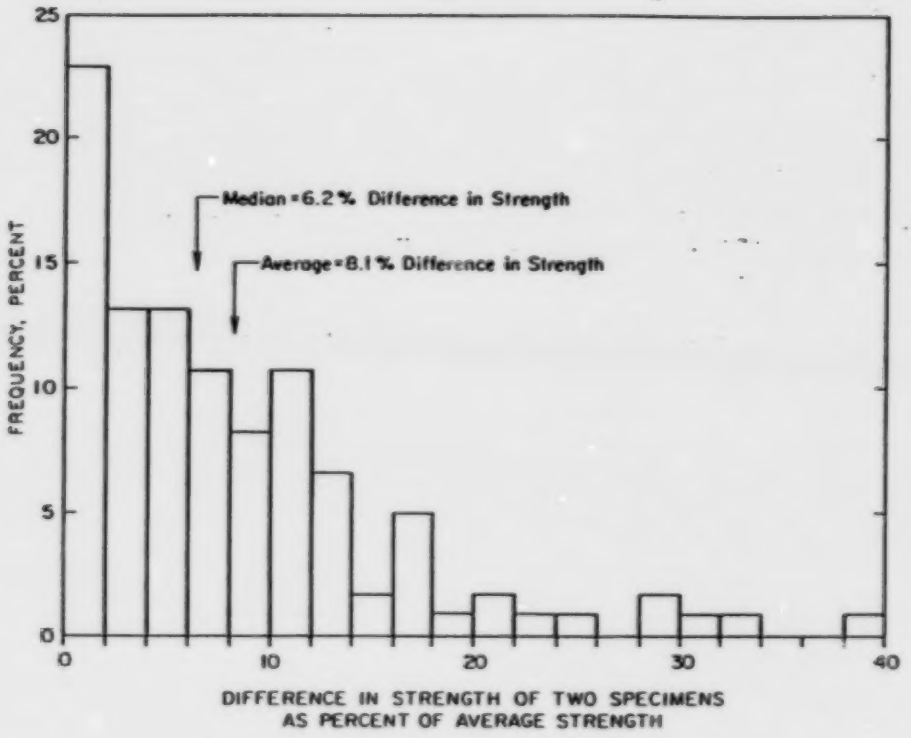

FIG. 1 Distribution of Variation of Test Results for 122 Sets of Duplicate Specimens

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